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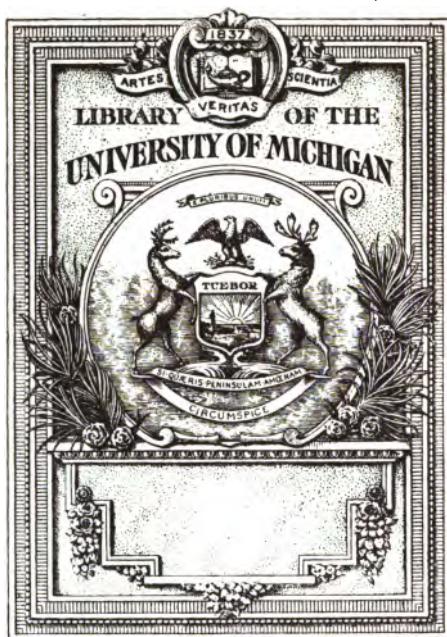
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**LABORATORY EXPERIMENTS**  
**IN**  
**GENERAL CHEMISTRY**

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*By*  
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Prof. Wm. Hale. 3-8-199.

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## PREFACE

The experiments outlined in this manual are designed primarily for college students who have not had a course in chemistry in a preparatory school. The exercises represent the laboratory work of a comparatively brief introductory course in General Chemistry. The experiments are to be conducted under the guidance of an instructor, and are to be supplemented by class room demonstrations in connection with recitations from a text-book in General Chemistry for college students, or by illustrated lectures and text-book assignments.

Frequently the experiments do not furnish sufficient information to enable the student to answer some of the questions and make the explanations that are to appear in his notebook. The necessary information can generally be obtained from the text-book, and it is my plan to have the student use the text-book and laboratory outline as companion volumes in the laboratory. To facilitate the student's progress in this connection page references to two widely used texts have been inserted. My experience goes to show that the student will thus make very efficient use of his time, that he will give care and thought to his work, and that the laboratory work can be successfully made the central feature of instruction in the course.

In preparing the experiments, the substances chosen for study have purposely been limited to avoid scattering the student's efforts. Intensive, rather than extensive, study has been the underlying idea in selecting the exercises. Extended experiments on the metallic elements have not been included, as I feel that this work should be undertaken in Analytical Chemistry, and be allotted some of the time so largely used for laboratory practice in following a scheme of separations. I fully realize the value of practice in the identification of "unknown" substances, and this feature of laboratory work has been duly emphasized, and, it is hoped, in a manner which preserves its educational value. Emphasis has also been

placed upon the general reactions of acids, bases, and salts, the processes of oxidation and reduction, and chemical changes prominent in everyday life. My experience has been that the work outlined is adequate in scope as a preparation for more advanced courses in chemistry, and that it offers the cultural benefits of laboratory work to the student who wishes to take only elementary chemistry as a part of his college course. The benefits of laboratory work, of course, lie more in the hands of the instructor than in the pages of the book, and fully as much in the enthusiasm and spirit of inquiry of the student as in textbook matter and qualities of the teacher. In the pages of this little book I have sought throughout to enforce the scientific method of work. The educational value of the experiments will be realized by the student in so far as he succeeds in making the scientific method a life habit.

The majority of the experiments have been collected from various sources and modified in some respects by substitutions and additions of original material. Helps and suggestions to the student have been distributed in the form of notes with a view of encouraging him in his efforts, and gradually building up higher standards and ideals. The optional experiments are designed for the faster workers in the class, and will serve a very useful purpose in keeping the ambitious student from getting too far ahead of the recitation work of the class. The interest of the bright, quick student often flags, and the quality of his work deteriorates because he reads ahead of the class, and then fails to prepare the regular assignment. The chapter of elective experiments will be found of excellent service in this connection.

I am indebted to the Laboratory Outline of Alexander Smith for many ideas in laboratory instruction. I also desire to express my thanks to Mr. E. E. Morlan of this University for helpful suggestions in the selection of material for the experiments. Corrections and suggestions will be appreciated.

HERMAN SCHLUNDT.

University of Missouri, August, 1910.

## GENERAL INSTRUCTIONS

1. Check the outfit of apparatus listed on the printed card in your locker. To do this, put all the articles on the top of the desk, then check and return such pieces as you can identify. Since articles missing, broken, or imperfect will be charged for when the course is completed, each piece as it is returned to the locker should be carefully examined, and any shortages or defective apparatus should be reported to an instructor. Place the test tubes in the rack provided for them, and arrange the glassware so that opening and closing the drawers will not break or damage the apparatus. Any unfamiliar articles may be checked with the aid of an instructor. Finally sign the card and hand it in at the store room.

2. Provide yourself with a notebook and make a careful permanent record of each experiment unless the directions state otherwise. Enter the numbers and titles of the experiments as they appear in this manual. Record what you observe in short, clear statements and at the time the observations are made. Having made a record of the observed facts, enter the conclusions you draw. Do not copy the directions, but direct your efforts to an intelligent interpretation of the data presented by the experiment, and then express your ideas in accurate terms.

3. Where an interrogation point, (?), or a direct question appears in the directions, a corresponding note should appear in the notebook. Oftentimes the experiment itself does not furnish sufficient data for answering some of the questions asked. The necessary information must then be sought by referring to a text-book. To save time in such cases page references to two well-known texts are given. K. refers to Kahlenberg's Outlines of Chemistry, and S. to Alexander Smith's General Chemistry for Colleges.

4. When the word (Instructions) appears, consult the instructor before going further.

5. The chemicals are divided into three sets, each arranged alphabetically according to the scientific names. The first set contains solids in bottles or jars, the second liquids in small bottles, and the third liquids in large bottles. The bottles and their places are numbered to facilitate correct replacement on the shelf, and particular care should be taken to return them to their proper places. Read the labels carefully, and you will not be using a sulphate where sulphite is required, or a concentrated acid where a dilute one should be employed.

6. All students work independently, except where the directions say "Two students working together."

# LABORATORY EXPERIMENTS IN GENERAL CHEMISTRY

## CHAPTER I.

### APPARATUS.

#### 1. The Bunsen Burner.

a. Unscrew the chimney and examine the construction of the burner. Make a drawing to scale showing the various parts.

**Note 1.** The purpose of drawings in the laboratory notebook is not to represent a picture of the apparatus, but to show its arrangement and operation. For this reason, and also for simplicity, sectional drawings or elevations are generally better than perspective effects. The student should aim at skill in making simple drawings rapidly and neatly with little or no use of a ruler.

b. Attach the burner by means of rubber tubing to the gas-tap, close the air-holes at the base, and light. Open the air-holes gradually and note the effect on the flame. (Size, shape, luminosity, steadiness.) Suggest a cause of the difference in the two flames. When the air-holes at the base of the burner are open the gas should burn with a noiseless blue flame.

c. Explore the non-luminous flame with a platinum wire to locate the relatively hotter and cooler parts. (?) Where should an object be held to get the greatest heating effect? Hold a match across the flame about 1 cm. above the top of the burner. (?) Show that unburnt gas exists in the inner cone by leading it out through a glass tube and lighting it. What region is deficient in air, and which has an excess? Make a scale drawing of the non-luminous flame showing its structure.

d. Try to vary the length of the flames. Can you obtain a luminous flame 2 cm. high? A non-luminous flame of the same height? How could the velocity of the gas in the chimney be changed without changing the pressure of the gas?

How would a change in the velocity of the chimney gases change the structure of the flame?

**Note 2.** Perhaps you will find it necessary to conduct further experiments before answering some of the foregoing questions. When the apparatus for such experiments is not in the desk outfit, it may generally be obtained from the store room on temporary order. The supply of apparatus given out on temporary order is usually limited. It should therefore be returned promptly, so as to make it available for other students. For some forms of apparatus the store-keeper will require an order from the instructor.

e. What volume of gas is consumed by an ordinary Bunsen burner per hour?

## 2. Capacities.

a. With a graduated cylinder measure 100 cc. of water and pour it into one of your beakers. Readings on the cylinder are taken at the bottom of the meniscus, and the line of sight should be in the same plane. Next estimate the capacity of each of your beakers, and record your estimate. Then measure the volume of the beaker, filling it to a convenient height for use and not to the brim. In the same way make estimates and find the volumes of several test tubes, the flasks, and bottles in your desk outfit of apparatus. Record the results in tabular form somewhat as follows:

**Capacities of Vessels.**

Name of vessel.	Estimated volume.	Measured volume.	Difference.
Beaker No. 1.	60 cc.	72 cc.	- 12 cc.
Beaker No. 2	125 cc.	115 cc.	+ 10 cc.

b. With the triangular file make a scratch on the neck of one of your flasks. Fill it to the mark with water and measure its volume with the graduated cylinder. Now weigh the flask on the platform scales. Again fill to the mark with water and weigh. Find the volume of the flask by the method of weighing. Refer to table 4, Appendix. Which value for the volume do you regard the more accurate, and why?

**3. The Wash-bottle. (Figure 1.) Its Construction and Use.**

a. Use a flask of about 500 cc. capacity, and a two-holed rubber stopper. Attach the nozzle with a short piece of rubber tubing.

**Working of Glass Tubing. Cutting.** To cut tubing of small size, make a scratch at the desired point with a triangular file. Hold the tubing with both hands, place the ends of the thumb nails together opposite the scratch, and press forward with the thumbs and pull back with the hands.

**Bending.** To bend glass tubing, heat in the ordinary gas jet, holding it lengthwise in the flame, and entirely in the luminous part, rotating it to get an even heating. Do not use the Bunsen flame unless the burner is provided with a wing-top. When the heated portion has become soft enough to bend by its own weight take the tubing from the flame and bend it at once to the desired angle. While bending, sight along the tube to aid you in making the two limbs come in the same plane.

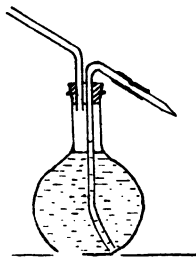


Fig. 1.

**Fire Polishing.** The sharp edges of tubes should be rounded off by heating the ends in the Bunsen flame.

**Drawing.** The nozzle is made by heating a portion of the tubing evenly in the Bunsen flame until it is softened, the end portions being held in the hands. Then take the tube from the flame and draw it out slowly. When cool cut the contracted part at the desired place and fire polish cautiously.

In fitting flasks with stoppers hold the flask firmly by the neck and not at the bottom. When inserting glass tubing in rubber stoppers or corks moisten the tubing or use a little

vaseline, and hold it near the end to be pushed into the stopper. Cuts and accidents too often result from faulty methods of work.

No record of this experiment is made in the notebook up to this point. Submit your finished apparatus to the instructor for criticism and approval. Finally fill the flask with distilled water and keep it in your locker ready to be used for washing, rinsing, and preparing solutions. Use tap water for other purposes.

b. **Filtering and Washing.** In the smallest flask or beaker place about 10 g. of common salt, and add about 30 cc. of distilled water. Shake the mixture to aid solution. Separate the liquid from the undissolved solid by filtering: Fold a circular sheet of filter paper in halves and then in quarters. Open the folded paper so that a cone is produced, and place the cone in a dry glass funnel. Holding the paper in position with one hand, blow a little distilled water on it from the wash-bottle and with the finger press the paper firmly to the sides of the funnel. Next place the funnel in a filter stand, the stem extending into and touching the side of a beaker or flask below. It is not advisable to use the retort stand for holding a funnel. Why is a wooden stand preferred? To avoid splashing in transferring to the filter, hold a glass rod against the rim of the beaker at the point where the liquid is to flow out. The liquid will generally follow the rod, and by resting the lower rounded end against the filter, the liquid can be transferred without loss or spattering.

Rinse the beaker with a jet of water from the wash-bottle, and when the liquid has run out wash the residue on the filter with a stream of water from the wash-bottle directed on the paper near its edge. Continue the washing until the taste of salt cannot be detected in a drop of the washings. Suggest a more delicate test for the presence of salt in the wash water. Try your test. (?) (S. 8.)

Evaporate a portion of the filtrate on a watch glass, or an evaporating dish, placed upon a beaker of boiling water. Examine the residue. Is it still common salt? In what respects does it differ from the original sample?



**Note 3.** Cleanliness and neatness in working are of the utmost importance. Care should be taken in little things. Attention should be given to the appearance of your apparatus. In setting up apparatus, ring-stands, filter stands, etc., should be placed straight on the desk. Iron rings, clamps, etc., which are not actually in use should be removed from the retort stand. Have the rod of the stands away from you, not toward you. Keep your apparatus clean and arranged in an orderly manner in your locker. If liquid has been spilt wipe it up directly. At the close of a laboratory period clear the desk and leave it clean and dry.

What is the size of the angle of the filter funnel?

#### **4. The Simple Balance.**

The laboratory is provided with several types of balances to meet the varied requirements of the experiments. At times the student may be uncertain as to which balance is best suited for the weighings to be made. A moment's reflection, however, will generally result in the proper choice. For example, when an accuracy in weighing of a tenth per cent is demanded, the weight of an object of 1000 g., when determined to within 1 g., meets the requirements. On the other hand, the weight of a 1 g. body must be determined to the nearest milligram, a thousandth gram, to be within the same limit of error,—a tenth of one per cent. In the former case a balance sensitive to a gram will answer the purpose; but for the latter the balance must be sensitive to a milligram. The platform scales will safely carry a load of 2 kilograms on each pan, and will thus serve very well for making weighings of relatively heavy forms of apparatus. The balance provided with a pointer should be used in quantitative experiments, where relatively small differences in weight are to be determined and the gross weight does not exceed 100 grams. The horn-pan balances will answer for some of the quantitative experiments and are convenient for weighing out approximate quantities of solid reagents. Look, before you leap: An attempt at weighing an object of a kilogram mass on a balance designed for a maximum load of 100 g. may ruin the balance and thus turn out an expensive experiment for the student.

**Directions for the Use and Care of a Balance.** (1) To release the beam and pans of the balance turn the screw or lever on the base of the balance in front.

(2) To determine the zero point, take a half of the total number of scale divisions passed over by the pointer in one passage; count off this number of divisions from either end of the swing, and use this point as the zero. The zero of any one balance changes, and must be redetermined every time a weighing is made. The zero is never read by waiting for the pointer to come to rest.

(3) Handle all weights with clean forceps; never with the fingers.

(4) Return the weights to their proper places in the box; never place them on the desk or the base of the balance.

(5) Always arrest the balance when not in use, and every time weights or other objects are added or removed from the pans. Never take off a weight whilst the beam is swinging.

(6) How sensitive is the balance? For an answer, place a 10 gram weight on each pan and note the zero as before. Now increase the weight on the right hand pan by 10 mg. As before, find the position at which the pointer would come to rest. The difference between the two points of rest of the pointer gives its deflection for 10 mg., i. e., the sensibility of the balance for a 10 g. load. This value may be used for determining weights less than 10 mg.

(7) Never weigh solid or liquid reagents directly on the balance pan. In case of a solid carefully weigh a dry watch glass (or a test tube) first, then place the substance upon the glass and weigh again, and take the difference as its weight. For a liquid a small beaker (or a test tube) is used instead of a watch glass.

(8) Check your count of weights by the places vacant in the box.

(9) Express weights in grams and decimals thereof. The weight of an object of 17 grams, 8 decigrams, and 3 centigrams is recorded, 17.83 g.

(10) Record the weights in the notebook, or the laboratory manual, never on loose sheets of paper.

(11) Be systematic in making weighings. Every change of a weight should bring you nearer to the final result. Cultivate the estimation of weights and volumes in the metric system.

(12) Weights are generally placed on the right hand pan.

#### Practice.

(1) Ascertain the weight of several objects supplied by the instructor. Weigh each separately and then find the combined weight by weighing. If the sum of the weights of the individual objects differs from the total weight found by more than 0.02 g. repeat the weighings.

(2) Weigh a five-cent piece.

(3) Set up a burette as shown in Fig. 33, K. Clean the burette. (Instructions). A clean burette will not leave streaks of water or drops on its walls when liquid is withdrawn. Fit it with a short piece of rubber tubing and glass nozzle. The flow of liquid is controlled by a pinch-cock or by placing a glass bead in the middle of the rubber tube. When using the burette make sure that the rubber tube and nozzle are free from air-bubbles, and there is no leak at the nozzle.

Weigh a small dry beaker. From a burette measure into the beaker about 20 cc. of distilled water. Make the readings on the burette carefully, observing the lower line of the meniscus, and estimating tenths of a division. Allow about a minute between burette readings for the water to drain down the wall. Note carefully the volume of water measured into the beaker, expressing it in cubic centimeters, e. g., 17.85 cc. Now weigh the beaker and contents. From the data obtained compute the weight of 1 cc. of water. Criticise the result.

Make a systematic record of the data:

Weight of beaker and water ..... = 33.47 g.

Weight of beaker empty ..... = 15.74 g.

Weight of water ..... = 17.73 g.

Volume of water ..... = 17.95 g.

Weight of 1 cc. of water ..... = 0.988 g.

Allow the beaker to stand uncovered for 20 minutes, then weigh again. Compute the percentage loss in weight. Mention three factors that influence the rate of evaporation of water from an open vessel.

**5. Identification of Substances.**

Bring specimens of charcoal, saltpetre (potassium nitrate), and roll sulphur on watch glasses from the side-shelf.

a. Examine the sulphur carefully, noting among its specific properties, color, odor, hardness, and taste. In testing the taste of a substance touch only a minute quantity to the tongue. Is sulphur soluble in water? Test its solubility in carbon disulphide, using a dry test tube. (Caution:—Carbon disulphide is very inflammable. Keep it away from the flame.) The relative solubility of sulphur in water and carbon disulphide can be judged by the eye. To ascertain whether sulphur is appreciably soluble in water filter the mixture after shaking well, catch a few drops on a watch glass, and evaporate on a water bath. If the mixture settles readily pour off a few drops of the clear liquid directly upon a watch glass and evaporate. Is the stain upon the watch glass any greater than the same quantity of solvent leaves? Heat a small piece of sulphur in a test tube. (?) Does it appear to melt at a lower temperature than potassium nitrate? Compare its melting point with that of potassium nitrate. (K. 178, 349; S. 249, 366). Place a small piece of sulphur in a deflagrating spoon and heat it in a Bunsen flame. (?) Does it take fire before it melts? When it begins to burn remove the spoon from the flame and with the hand waft some of the gas formed toward the nose to test its odor.

Examine the specimens of charcoal and saltpetre, making similar tests and finally tabulate the properties of the three substances. Include in this table some other characteristic properties of these substances, such as the melting points, boiling points, densities, specific heats, electrical conductivity. (K. 214, 348, 178; S. 318, 365, 248.)

b. Gunpowder is made from charcoal, saltpetre, and sulphur. Place about 2 g. of the ordinary powder in a test tube, add 10 cc. of water, shake well, and also warm gently. Filter; wash the residue on the paper with a jet of water blown from the wash-bottle. Evaporate the filtrate on a water bath. Examine the residue and identify it. Dry the filter paper and its black residue by spreading it out on the sand bath and warming gently. Transfer the residue to a dry test tube and shake

it with about 3 cc. of carbon disulphide. Filter and allow the filtrate to evaporate without heating it. Describe and name the residue. Suggest a reason for drying the residue before shaking it with carbon disulphide. Examine the black residue on the paper and compare its properties with those of finely powdered charcoal, obtained by grinding a piece of charcoal in the mortar. What is the probable cause for any difference in the behavior of the residue and the powdered charcoal?

Does any chemical change occur during the manufacture of gunpowder?

Estimate the relative quantities of the components present in the specimen of gunpowder studied, and make comparisons with the composition of the ordinary powder. (K. 349; S. 366.)

## CHAPTER II.

### HYDROGEN.

**Note 4.** Before commencing an experiment read carefully the directions to grasp the purpose of the experiment, and to plan the work involved. Record in your notebook what you observe and guard against entering notes about what you think you should observe. If you find that your observations do not seem to be in accord with the text-book or the statements of the lectures, read the directions again, reflect a moment, and then try to clear up the discrepancies that appear to exist by repeating the experiment, and finally consulting the instructor. In this way, the scientific method of dealing with the problem in hand will be acquired, and the final solution of the difficulty with its thrill of satisfaction may prove of more educational value than several experiments without complications.

#### 6. Interaction of Metals and Acids.

a. Set up about a dozen test tubes in the test tube rack. Place a few pieces of each of the following metals in separate tubes:—Aluminium (turnings), copper (chips), iron (filings), lead (clippings), magnesium (wire or ribbon), platinum (wire), tin (granulated), zinc (gran.), zinc (dust). Pour into the graduated cylinder 25 cc. of concentrated hydrochloric acid and dilute it with an equal volume of water, stirring the mixture to insure thorough mixing. Add 5 cc. of the diluted acid to one of the metals, and watch the action carefully, and record your observations. If little or no action occurs in the cold, heat in a small Bunsen flame. (?) Follow the same method of experimenting with the other metals. When gas arises from the mixture, note where it first appears. Test the issuing gas by bringing a light to the mouth of the test tube. If a pop follows or a flame is seen, the gas is hydrogen. When the interaction is slight the gas set free may not test for hydrogen by this method. In such cases examine the gas by collecting a sample of it in a test tube filled with water and inverted over water, as shown in Fig. 2. The whole ap-

paratus may be conveniently mounted on a ring-stand. If the gas sample thus collected appears to be hydrogen give a reason for not detecting it as it escaped from the open tube.

Arrange the metals in the order of activity. In what respects does your order differ from the electrochemical series? (K. 435; S. 245.) Suggest a reason for some of the differences. Where would you place hydrogen in this series?

**Note 5.** Throw matches, wet filter papers and other solid objects in the waste-jars under the desk. Do not throw them into the sink.

What percentage (approximate) of the dilute acid used is water? (K. 50.) Assuming that water consists of 11.14 per cent hydrogen, and 88.86 per cent oxygen, and that hydrogen chloride consists of 2.77 per cent hydrogen and 97.23 per cent chlorine, compute the percentage of hydrogen in the acid. Is all of the hydrogen of the acid displaced in the course of the interaction with an excess of zinc? If not, formulate your ideas about the proportion of the total hydrogen that may be displaced from dilute acid by zinc.

b. Prepare some dilute sulphuric acid by pouring 5 cc. of the concentrated acid into 15 cc. of water. Try its interaction with small pieces of zinc and copper, in separate test tubes.

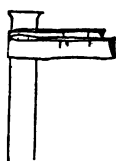


Fig. 3.

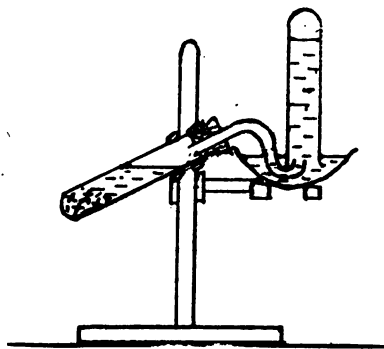


Fig. 2.

(?) Just cover a few pieces of zinc in a test tube with concentrated sulphuric acid. (?) Make a paper holder for the test tube and heat the mixture. (?) Test the odor of the products. (Exp. 5a.) What products of the reaction can you identify. Describe one of the products that is new to you. Try also the ac-

tion of dilute and concentrated sulphuric acid on pieces of copper.

c. Try the action of nitric acid, concentrated and dilute, on several of the metals, zinc or copper, and iron (wire). (?) Is hydrogen obtained? Exercise care in working with nitric acid. It leaves yellow stains on the hands.

d. Try the action of acetic acid on zinc and magnesium. (?)

e. Fuse the end of your platinum wire into a short piece of glass rod. Wrap the wire around a piece of zinc, and dip it into very dilute sulphuric acid (1:20). Do any gas bubbles appear on the wire? Is it dissolving? What part does the platinum wire take in this reaction? (S. 66.) Does gas form on the wire (a) when the zinc and platinum do not touch each other? (b) when the ends outside the acid are in contact?

What is the commercial name of hydrochloric acid? Of sulphuric acid?

Modify the experiment by using copper, carbon, or gold instead of the platinum wire. (?)

**Note 6.** When acid gets upon the clothing, apply ammonium hydroxide at once. Burns should be rubbed with a paste of sodium hydrogen carbonate (baking soda) and water. It is well to dress painful burns with a preparation of lime water and sweet oil. Cuts should be washed at once with running water, and dressed with boric acid, or a solution of lysol. Obtain the assistance of an instructor in case of injury.

### 7. Preparation and Properties of Hydrogen.

a. Fit a 250 cc. flask with a thistle tube and a delivery tube. Put 30-50 g. of granulated zinc in the flask and pour 50 cc. of dilute hydrochloric acid (1:4) on it. Collect some of the gas issuing from the delivery tube over water in the pneumatic trough (K. 16; S. 48). Test the gas to see whether it is free from air (source?) by bringing a light near the mouth of the test tube. Continue the tests with fresh samples until the gas in the test tube burns quietly.

Fill one of the wide-mouth bottles with hydrogen, and holding it mouth

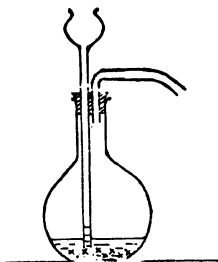


Fig. 4.



downward, thrust into the flask a burning splinter. Does the splinter continue to burn? Explain.

Show by an experiment that hydrogen is lighter than air. (?)

Cover a wide-mouth bottle filled with hydrogen with a dry sheet of filter paper, and hold it mouth downward, for two minutes. Then remove the paper and bring it quickly near a flame. (?) Try the experiment substituting a wet sheet of the paper. (?) Try it also with a sheet of note-paper. (?) Explain the results.

Replace the delivery tube with a glass nozzle. If the action in the generator has slackened pour 5 cc. of concentrated hydrochloric acid through the thistle tube. Hold a cold, dry beaker against the nozzle. If moisture is deposited what is its probable source? Connect a U-tube filled with fused calcium chloride between the nozzle and the outlet tube. Hold the cold beaker against the jet once more. (?) When the issuing gas is free from moisture test it to see that it is not explosive. Then light the jet and observe the color of the flame. (?) Why is the hydrogen dried for this experiment?

Burn dried illuminating gas at the nozzle, and hold a dry, cold beaker over the flame which should be small. (?) Explain.

Draw from memory a figure of the Kipp apparatus used in the class-room for preparing hydrogen.

b. (Two students working together). Fit a hard glass tube at least 20 cm. long with corks and tubes as shown in the accompanying figure. Dry specimens of ferric oxide, silicon dioxide (sand), and cupric oxide by heating in a crucible. Put one of the oxides in a porcelain boat and place it in the tube. Now connect the tube to a source of dry hydrogen and pass a stream of the gas through the tube. Test the issuing gas to see that it is free from air. Then heat the tube, cau-

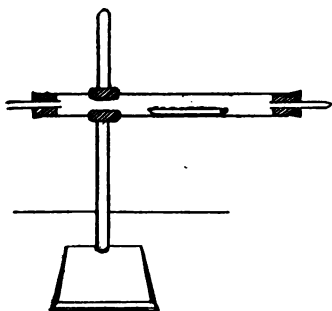


Fig. 5.

tiously at first by moving the flame back and forth, and later, strongly until the boat is red hot. What evidence does the experiment furnish that a chemical change is in progress during the heating?

Conduct similar experiments with the other oxides. Name the new products formed.

What property of hydrogen do these experiments illustrate? Assign a title to this paragraph in your notebook.

## CHAPTER III.

### OXYGEN.

#### 8. Sources.

Oxygen is one of the constituents of the following substances and the percentages show the approximate quantity present in each:

Lead dioxide, 13 per cent.

Potassium nitrate, 47 per cent.

Silicon dioxide (sand), 53 per cent.

Barium peroxide, 19 per cent.

Sugar, 51 per cent.

Manganese dioxide, 36 per cent.

Calcium carbonate (marble or limestone), 48 per cent.

Heat 2-3 g. of each of these substances in a dry, hard glass test tube. Observe whether gas is given off and in each case test it by inserting into the test tube a glowing splinter. Try the blast-lamp if the Bunsen flame fails to give results. If drops of liquid collect on the sides of the test tube during heating hold it in a nearly horizontal position. Why? Make a record of the changes noted in the course of the heating. Describe the residues. Arrange the substances studied in the order of their stability. (S. 81.)

**Note 7.** To dry test tubes quickly warm them in the flame, and then blow or draw a stream of air into the tube through a glass tube reaching nearly to the bottom.

To repair a test tube seal on a short glass rod in the blast-lamp. (Instructions.) Now heat the tube evenly a little above this joint. When the glass has softened pull off the end part. Soften the sealed end of the tube again, remove it from the flame, and holding it vertically, blow to round out the end. Repeat the softening and blowing until the wall is of nearly uniform thickness. Some persons prefer to heat the broken end of the tube directly in the flame until it closes up. The lump of glass in the bottom is then blown out as just stated. Test tubes of soft glass are readily repaired. Hard glass is more difficult to work.

### 9. Preparation and Properties.

a. Mix on paper about 5 g. of potassium chlorate and 3 g. of manganese dioxide. Place the mixture in a test tube fitted with a one-hole cork and delivery tube. Test the apparatus to see that it is air-tight. Then arrange the parts as shown in Fig. 6. Evolve the oxygen moderately slowly by regulating

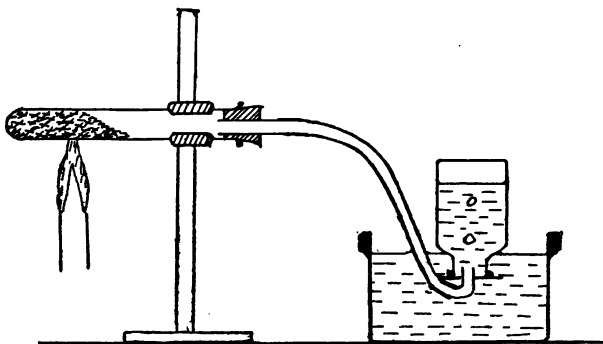


Fig. 6.

the heating. Collect the gas over water, filling five bottles. Keep the residue in the test tube and treat it as directed under c.

b. Test the odor of the gas. Inhale some of it. (?)

Place a small piece of sulphur in a deflagrating spoon, and burn it in one of the bottles of gas. (?) Identify the product by its odor. (Exp. 5a.)

Pour a little water into the bottle, close the mouth with the hand and shake. (Object of this?) Test the liquid with litmus solution or blue litmus paper. (?)

What is an indicator? (K. 130; S. 242.)

Burn a little red phosphorus in another bottle, and make the litmus test. (If yellow phosphorus is used, GREAT CARE must be taken not to get it on the hands. It catches fire easily, and causes severe burns. Cut it under water and handle it with forceps.)

Lower a piece of glowing charcoal into a bottle of the gas. (?) When burning has ceased, add a little distilled water (why not tap water?), cover the bottle with a glass plate and

shake. Add some clear lime-water, and shake again. Interpret the lime-water test. What is the chemical name of lime-water?

Repeat the burning of charcoal and make the litmus test. (?)

c. Add distilled water to the black residue in the test tube, and transfer it to a filter. Test the filtrate with a few drops of silver nitrate solution. Similarly, test small quantities of solutions of potassium chlorate and potassium chloride. (?) What inference may be drawn from these tests?

Wash the residue on the filter until a few drops of the washings produce little or no turbidity with a few drops of silver nitrate solution. Now dry the residue. Heat it in hard glass test tube in the blast-lamp and try the glowing splinter test. (?) If oxygen appears what is its probable source?

#### 10. Weight of a Liter of Oxygen. (Quant.)\*

In this experiment oxygen is evolved by heating potassium chlorate. Powder some of the chlorate and dry it on a watch glass by gentle heating on the sand bath. Weigh a hard glass test tube and place in it about 1.5 g. of the dried chlorate. Weigh the tube and chlorate together. Fit up the apparatus shown in Fig. 7, using the bottle of about one liter capacity

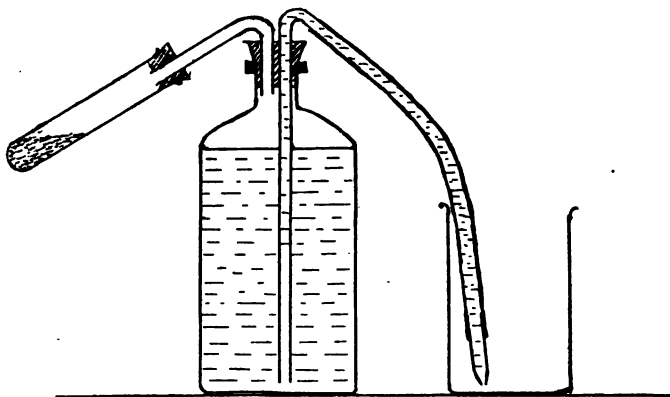


Fig. 7.

\*In experiments marked (Quant.) make the weighings on the finer balances provided with a pointer and scale.

for the aspirator. All joints must be perfectly air-tight. This can be ascertained by having the delivery tube leading to the beaker full of water and then opening the clip. If the apparatus is air-tight only a few drops of water will flow out at first. If it is not air-tight water will continue to trickle out. At the beginning of the experiment have the exit tube full of water and the aspirator at least two-thirds full. Allow the exit tube to drop to the bottom of the beaker, which should contain a little water and be of from 400-500 cc. capacity. Open the clip and by raising the beaker equalize the levels of the water in the aspirator and the beaker. (Why?) Close the clip; empty the beaker and then replace it. After opening the clip again, heat the test tube slowly, gradually raising the temperature. In the course of the decomposition of the chlorate smoke is oftentimes seen. This consists of solid particles and its loss from the tube should be guarded against. (Why?) If the test tube is of a rather small bore be watchful lest the passage for the gas becomes stopped. By heating near the upper level of the salt a free passage for the gas generally remains. When 300-500 cc. of the water has been driven out of the aspirator into the beaker, stop heating, allow to cool to the temperature of the room, equalize the levels and close the clip. Determine the volume of the water. (See Exp. 2b.) Weigh the test tube with the residue in it. Read the barometer, the temperature near the barometer, and the temperature of the water. You now have the weight of a known volume of oxygen standing over water at the temperature and barometric pressure observed. Reduce the volume to  $0^{\circ}$  and 760 mm. pressure, allowing on the corrected barometric pressure for the tension of water vapor. (Appendix, tables 5, 6 and 7.) Calculate the weight of a liter of oxygen at standard temperature and pressure.

What is the volume occupied by 32 g. of oxygen?

Should account be taken of the volume of the air originally present in the aspirator?

Is the apparatus of this experiment suitable for determining the weight of a liter of hydrogen? What changes in the apparatus would you introduce and why?

## Data.

Wt. of tube with chlorate .....	g.
Wt. of tube and residue .....	
<hr/>	
Wt. of oxygen .....	g.
Wt. of beaker with water .....	g.
Wt. of beaker .....	
<hr/>	
Wt. of water .....	g.
Temp. of water .....	
Temp. near barometer .....	
Barometric reading .....	mm.
Correction .....	
Barometer (corr.) .....	
Tension of water vapor .....	
<hr/>	
Partial pressure of oxygen .....	mm.

11. Melt a small quantity of potassium chlorate cautiously in a test tube clamped in a vertical position. Use a very small Bunsen flame for heating. Can the chlorate be melted without decomposing? Throw a pinch of manganese dioxide into the molten chlorate. (?) Apply the glowing splinter test. Interpret the result. (S. 54.) Assign a name to this experiment in your record.

## CHAPTER IV.

### WATER.

#### 12. Purity.

a. Taste some distilled water. Could you distinguish it from tap water by this property? Suggest two other methods of distinguishing it from tap water. Try your methods. Devise a method of testing distilled water for the presence of dissolved air, and suggest a method of removing the air dissolved in it. Compare the tints produced on litmus paper respectively by distilled and tap water.

b. **Union with Oxides.** In separate test tubes place small quantities of the following oxides: Cupric oxide, quick-lime, ferric oxide, phosphorus pentoxide, barium oxide or peroxide. Add a few cc. of distilled water to each and shake. Filter and test the filtrates with litmus solution or litmus paper. (?)

What other acid forming oxides have been examined in earlier experiments?

What class of elements furnish acid forming oxides? (K. 125; S. 81.)

#### 13. Hydrates.

a. Gently heat specimens of barium chloride, Glauber's salt, potassium nitrate, alum, potassium dichromate, and sugar. Note the results that occur during the course of the heating and record the results in tabular form, under the following headings:

Substance heated.	Changes during heating.	Amount of deposit in cool portion of tube.	Appearance of residue.
-------------------	-------------------------	--------------------------------------------	------------------------

Are all crystalline substances hydrates? Which of the substances examined would you classify as hydrates? Does sugar lack any of the characteristics of hydrates? (S. 82; K. 257.)



b. Place several small crystals of blue vitriol in a test tube, cover them with concentrated sulphuric acid, and note the changes that occur in the course of a half hour. Meanwhile heat several crystals of blue vitriol in an evaporating dish. Set aside a small portion of the resulting white powder on a watch glass. (?) Boil the remainder with sufficient water to dissolve it. Avoid an excess of water. Set the solution aside to cool. (?) Drain off the sulphuric acid from the crystals and add a few drops of water to the residue in the test tube. (?)

c. (Optional; Quant.) Weigh out accurately, about 2 g. of gypsum in a porcelain crucible. Place the crucible on a clay triangle, and heat to redness. When cool, weigh, and then heat a second time and weigh. When the weight is constant calculate the percentage loss in weight. Record the data in systematic form as suggested by the following outline:

**Hydrate: Gypsum.**

Wt. of crucible with gypsum.	= 9.485	g.
Wt. of crucible	= 7.620	g.
	<hr/>	
Wt. of salt taken	1.865	g.
	<hr/>	
Wt. of crucible with residue	=	g. (First weighing)
Wt. of crucible with residue	=	(Second weighing)
Wt. of crucible with residue	=	(Third weighing)
Wt. of crucible	=	
	<hr/>	
Wt. of residue	=	g.
Loss in weight	=	g.
Percentage loss in weight	.....per cent.	

d. (Quant.; optional.) Weigh a crucible with cover. Place in the crucible about 2 g. of blue vitriol. Crystals having a white crust should be rejected. Cover and weigh. Now support the covered crucible over a Bunsen flame so that the top of the flame comes within 3 cm. of the bottom of the crucible. Heat thus for ten minutes. Allow to cool; and then

weigh. Heat for five minutes longer; weigh again when cool. When no further change in weight occurs, use the data to calculate the percentage loss in weight. What does the loss in weight represent?

Suggest a reason for not heating the blue vitriol as highly as the gypsum in these experiments.

#### 14. Solubility. (Quant.)

Pulverize about 15 g. of potassium dichromate. Prepare a saturated solution of the substance by shaking (Patience!) it in a stoppered flask with 50 cc. of water. Take the final temperature. Now decant the clear solution into a burette, and measure a definite volume, 20-30 cc., into a weighed evaporating dish, and weigh again. Evaporate the solution on a water bath, completing the final drying by gentle warming over a free flame. (Instructions.) Weigh the residue, and from the data in hand compute the quantity of the salt that would be dissolved by 100 g. of water at the observed temperature. Calculate also the quantity of salt that a liter of this solution would contain. Make a tabulated statement of your data, and record the solubility determinations of three other students. Compare the results with a view of accounting for some of the differences in the values as compared with your result. What was the density of your solution?

## CHAPTER V.

### EQUIVALENT WEIGHTS, FORMULAS, EQUATIONS.

#### 15. Composition of Magnesium Oxide. (Quant.)

Weigh a crucible with cover. Clean a piece of magnesium ribbon about a meter long with a bit of emery cloth. If the magnesium is bright it may be used without cleaning. Roll the ribbon up loosely but small enough to lie in the crucible. Place it in the crucible, cover and reweigh. Place the crucible and contents on a triangle and heat it at some point where the magnesium touches it. As soon as the magnesium begins to glow regulate the supply of air by sliding the cover on and off. Allow as much air to enter as is possible without letting the white oxide escape. A little loss will undoubtedly occur, but great care should be taken to have the loss small. After the burning has nearly ceased, remove the cover entirely, but guard against the loss of oxide that clings to it. Now heat the crucible to bright redness for five minutes in the top of the flame. Toward the end of the heating the solid may be carefully stirred with a pointed glass rod. It is well to heat the rod to keep it from breaking when it touches the hot oxide. What is the necessity of stirring the powder? Allow the crucible to cool and then weigh. Continue the heating, cooling and weighing until a constant weight is obtained. The contents of the crucible should finally be perfectly white. Interpret the gain in weight.

From your data calculate the weight of magnesium combining with 8 parts of oxygen.

Wt. of Ox. found : Wt. of Mg. taken : 8 : x.

This gives x, the equivalent weight of magnesium.

Record the data obtained in the experiment in tabular form as follows:

Wt. of magnesium and crucible.....	=	g.
Wt. of crucible .....		
Wt. of magnesium .....		
Wt. after heating (1) .....		

Wt. after heating (2) .....  
Wt. after heating (3) .....  
Gain in weight .....  
Per cent of gain .....  
Eq. Wt. of magnesium .....

Compare your result with the values obtained by two other students for the equivalent weight of magnesium.

Assuming the atomic weights of magnesium and oxygen (K. 81; S. inside rear cover) calculate from your data the formula for the oxide of magnesium.

#### 16. Composition of an oxide of iron. (Quant.)

Weigh an evaporating dish, and place in it about 1.5-2.0 grams of bright iron wire, and weigh again. Cover the dish with a watch glass, convex side down (why convex side down?) and add 20 cc. of dilute nitric acid. Heat on the water bath, and when all the iron has dissolved rinse the watch glass and remove it. Evaporate the solution to dryness on a beaker of boiling water. Then place the dish on a triangle supported on a ring-stand and heat carefully with a burner held in the hand. (Instructions.) After red fumes cease to be given off, heat the residue to redness. Allow to cool and weigh. Finally, heat to constant weight.

When the red gases are set free, the nitrate of iron is decomposing. The red fumes show the presence of one of the oxides of nitrogen in the gaseous products. The residue is an oxide of iron. The increase in weight represents the oxygen combined with the iron.

From the data obtained, calculate the equivalent weight of iron.

Assuming the atomic weights of iron and oxygen, calculate from your data the formula for the oxide of iron. What is the name of this oxide?

Magnesium, zinc, tin or copper, may be used instead of iron, and the method of operation is essentially the same. The residues, however, in some cases, cannot be dried on the water bath. Extra caution must therefore be exercised in heating them to avoid loss by spattering.

N. B. Whilst the solution is evaporating the next experiment may be performed. Regulate the heating by lowering

the flame of the burner so that the water boils off slowly, and then proceed with other work. Waiting around for evaporations may be good training in the cultivation of patience, but evidently such time is not to be credited as laboratory work.

**17. Equivalent Weight of Zinc by Displacing Hydrogen. (Quant.)**

a. First fill the pneumatic trough and 1-l. bottle with water so that the liquid may acquire the temperature of the room. Fit a 100 cc. flask with a drop-funnel and a delivery tube as shown in Fig. 8. Test the apparatus to see that it is air-

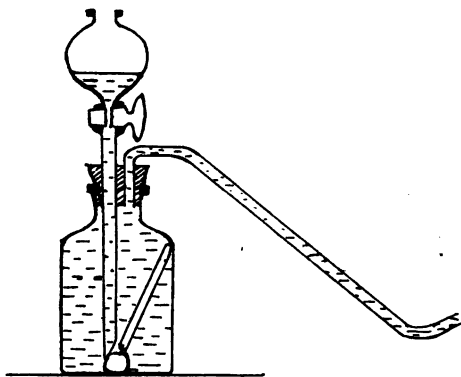


Fig. 8.

tight. Weigh a piece of pure zinc (Storeroom) of about 2 g. Without detaching the platinum wire from the glass rod, wrap it around the zinc (why?) and slip the whole into the flask. Now fill the apparatus completely from the stop-cock of the drop-funnel to the tip of the delivery tube with water. Invert the liter bottle with water in the pneumatic trough, and slip the end of the delivery tube into the mouth of the bottle.

Fill the bulb of the funnel with pure concentrated hydrochloric acid and admit this to the flask, in such a way that a steady stream of gas flows into the collecting bottle. When the metal is entirely dissolved, drive all gas over into the bottle by pouring water through the funnel.

The weight of the hydrogen displaced in this experiment is not to be ascertained by direct weighing, but by determining the volume of the gas and calculating its weight from this and the density of the gas. Proceed as follows: After the gas has assumed room temperature, equalize the levels of the water inside and outside the bottle. While in this position cork the bottle and remove it from the trough. Find the volume of the gas. (See Exp. 2b.) Record the temperature of the water in the bottle. Read the barometer and the temperature near it. Since the hydrogen is mixed with water vapor, correct for the latter (Exp. 10) and then reduce the volume of the hydrogen, by rule, to standard conditions. From the data obtained, calculate the equivalent weight of zinc, i. e., the quantity of zinc which displaces 1.008 g. of hydrogen:

Wt. of hydrogen : Wt. of zinc : 1.008 : x.

What is the valence of zinc?

How many molecular weights of hydrogen chloride are required to furnish the hydrogen that one atomic weight of zinc will displace? Write the part of the equation that expresses your answer to this question.

b. Determine the hydrogen equivalent of some other metal by the above method,—magnesium, using 0.7 to 1.0 g.; aluminum, 0.5 to 0.8 g.; or iron, 1.5 to 2.0 g. Whilst the zinc is dissolving, clean and weigh out the other metals.

Refer to the experiment, "Weight of a Liter of Oxygen" and use the plan outlined there in recording your experimental data.

#### **18. Composition of Lead Chloride. (Quant. Optional.)**

Weigh two evaporating dishes. In each place about 1 to 1.5 g. of sheet lead and weigh again. Dissolve the lead in nitric acid diluted in the proportion of one volume of acid to 2 volumes of distilled water. About 25 cc. of the diluted acid will be required for each sample. Heat the materials on a water bath to hasten the reaction and keep the dishes covered with watch glasses during this operation. When the lead samples are entirely dissolved rinse the covers, and remove them. Then add to each dish about 10 cc. of pure concentrated hydrochloric acid. Evaporate the contents of the dishes to

dryness on the water bath. Moisten the residues with about 2 cc. of acid and dry once more. Now heat the residues cautiously with the Bunsen flame and allow them to cool; then weigh. Interpret the increase in weight.

Record the experimental data as follows:

Experimental Data	Dish 1.	Dish 2.
Wt. of lead + dish .....		g. g.
Wt. of dish .....		
Wt. of lead .....		
Wt. of dish + lead chloride .....		
Wt. of dish + lead .....		
Wt. of chlorine .....		
Eq. wt. of lead .....		

Assuming the equivalent weight of chlorine to be 35.46, calculate the equivalent weight of lead.

Compute the percentage composition of lead chloride.

Assuming the atomic weights of lead and chlorine deduce a formula for the chloride of lead.

When lead dissolves in nitric acid a solution of lead nitrate is obtained. Write an equation for the interaction of lead nitrate solution and hydrochloric acid.

Save the lead chloride obtained for use in Exp. 23.

#### 19. Combining Weights of Zinc and Chlorine. (Quant.)

Weigh an evaporating dish and place in it about 2 g. of pure zinc (Storeroom) and weigh again. Wrap the platinum wire around the zinc, and then dissolve it in diluted hydrochloric acid (1:2), keeping the dish covered with a watch glass during the operation. Remove the glass and wire, rinse both, and evaporate the solution on the water bath as far as possible. Place the dish next on a triangle and evaporate it slowly to dryness. Then heat the white residue to the point where it has melted and no further. When the dish has cooled so that it can be borne on the hand, weigh it quickly. Repeat the melting, cooling and weighing, and take the lower result as correct.

Calculate, from the data obtained, how much chlorine combines with the equivalent weight of zinc found in the previous experiment. This amount is the equivalent weight of chlorine,  $x$ ,—

Wt. of zinc : Wt. of chlor. : Equiv. of zinc : x.

Assuming the atomic weights of zinc and chlorine, determine the formula of the compound.

Express the whole reaction of zinc and hydrochloric acid by making the equation in accordance with the results obtained in this and Exp. 17. Which of the factors in the equation have you determined experimentally, and which not? What law do we use in assuming that the undetermined factors are correct?

Record your data in tabular form.

## 20. Law of Multiple Proportions. . (Quant.)

Dry samples of potassium chlorate and potassium perchlorate on watch glasses by gently warming on a sand bath, or radiator. Weigh two hard glass test tubes, and introduce about 1 g. of the chlorate in one tube and weigh again. In the other tube place about the same quantity of perchlorate and weigh. Now heat the tubes cautiously and decompose the compounds, allowing the oxygen to escape. Should white vapors—a cloud of solid particles—appear at the mouth of the tube the heating must be slackened for a time. When the decomposition is complete (test ?) allow the tubes to cool and weigh the tubes with their residues.

Record your weighings as suggested by the following outline:

	Chlorate.	Perchlorate.
Weight of tube + salt.....	g.	g.
Weight of tube ..		
Weight of salt taken .....		
Weight of tube + residue.....	g.	g.
Weight of tube .....		
Weight of residue .....		
Loss in weight of salt .....		

Assuming that the loss in weight in these experiments represents the oxygen in the compound, calculate how much oxygen is combined with one gram of each of the residues. Assuming that the residues are the same substance, potassium chloride, show how the data just computed illustrate the law of multiple proportions.



Assuming that the formula for potassium chloride is  $KCl$ , and its molecular weight 74.6, calculate the quantities of oxygen combined with 74.6 parts of the chloride in each of the above experiments:

Wt. of chloride : Wt. of oxygen : 74.6 :  $x$ .

Where  $x$  is the weight of oxygen combined with one molecular weight of potassium chloride in each experiment.

Taking the atomic weight of oxygen 16, deduce the formula for each of the substances from the data now in hand, and finally write equations that will express the quantitative relations here determined.

21. Law of Multiple Proportions. (Quant., optional, two students working together.)

Fit a piece of hard glass tubing at least 20 cm. long with glass tubes and corks as shown in Fig. 5, and mount the tube for heating with the Bunsen flame. Make sure that the apparatus is air-tight. On the radiator dry specimens of pure litharge and lead dioxide. Weigh two porcelain boats and fill the boats with litharge and the dioxide respectively, using at least 2 g. of each, and then weigh again.

Place the boats in the tube of hard glass so that the points of the boats touch in the center of the tube. Connect the tube with a supply of dry hydrogen or illuminating gas and pass a gentle stream of the gas through the tube. Test the issuing gas to see that it is free from air. Now heat the boats moderately waving the flame to and fro at first. What collects in the cooler part of the tube? Where does it come from? When minute metallic globules appear at any point in the boats, withdraw the heat from that part of the tube for a time. The residue is more easily removed from the boats afterward if it does not fuse. After ten minutes of heating, remove the flame and allow the boats to cool in a stream of hydrogen. Remove the boats and weigh them. Reheat them until constant weight is obtained.

Record the data so that the weighings for the oxides will appear in parallel columns.

The loss in weight represents the oxygen removed from the oxides. Calculate the quantities of lead in combination with

8 g. of oxygen in the oxides and apply the law of multiple proportions to the data thus obtained.

**22. Law of Dulong and Petit.** Formulate the law.

Take the equivalent weights you have found experimentally in this chapter, viz.: magnesium, lead, zinc, iron, aluminum, etc., and multiply each by the corresponding specific heat. (K. 78; S. 135.) If the product is about 6.4, the atomic weight is the same as the equivalent weight. If not, then the smallest multiple that will bring the product up to about 6.4 is the valence of the element, and the product of this integer and the equivalent weight is the atomic weight of the element.

Arrange your data in the form of a table under the following heads:

Element	Equiv. Wt. found.	Specific heat.	Eq. Wt. $\times$ Sp. Ht.	Valence	Atomic weight.	Atomic heat.
---------	----------------------	-------------------	-----------------------------	---------	-------------------	-----------------

Plot the specific heats, K. 78, or S. 135, against the atomic weights and name the curve obtained by connecting the points.

Compare this curve with the one obtained by plotting the pressures against corresponding volumes for a given volume of gas, the temperature remaining constant (Boyle's Law). What is the equation for the curve?

## CHAPTER VI.

### REVIEW EXERCISES.

a. Referring to your laboratory notebook, consider the chemical changes involved in the experiments that you have performed, and write equations for the reactions. In several of the experiments nitric acid was employed, and since the reactions of metals and nitric acid are complex, the nature of the products depending upon the concentration of the acid and the temperature, the equations for these changes need not be attempted.

b. What exceptions do your experiments furnish to the statement that hydrogen is obtained by the reaction of acids with certain metals, e. g., zinc or magnesium.

c. Experiment 11 is often cited as an illustration of catalytic action, and manganese dioxide is referred to as a catalytic agent. In what other experiments did you employ catalytic agents?

d. Calculate the molar solubility of potassium dichromate from the data of experiment 14.

e. What is the mineralogical name for the oxide of iron obtained in experiment 16?

f. Give a reason for weighing the sample of zinc chloride obtained in experiment 19 while it was still warm.

g. Criticize the statement that the vapor tension of water is directly proportional to the temperature. (Appendix 6.)

h. Compare the volume of the oxygen as measured when standing over water as in experiment 10, **Weight of a Liter of Oxygen**, with its volume if measured over mercury under the same conditions of temperature and pressure.

i. What law is illustrated by the results of experiment 18, **Composition of Lead Chloride**?

j. How much zinc chloride will be furnished by dissolving 25 g. of pure zinc in pure hydrochloric acid? Use the data that you obtained in experiment 17. Calculate the quantity of the

chloride from 25 g. of zinc using the equation that you have written.

k. What chemical test would establish the fact that the residue left when potassium chlorate and perchlorate are decomposed, experiment 20, consists of a chloride?

l. Criticize the statement that the amount of oxygen in lead dioxide is twice that of the monoxide. (Experiment 21.)

m. Calculate the number of calories required to raise the temperature of 207 g. of lead  $1^{\circ}$ . What term in the table of experiment 22 properly designates this value?

n. Assuming the molecular weights of cupric sulphate and water, deduce a formula for blue vitriol from the data of experiment 13d.

o. Make a similar calculation with the data of experiment 15c.

## CHAPTER VII.

### THE HALOGENS.

**Note 8.** Read carefully the direction before beginning an experiment. The absence of italics and black faced type means that every line is significant. If you grasp the purpose of the experiment at the outset and then follow the outline thoughtfully your daily achievements will make steady gains. Hasty work and makeshift mounting of apparatus generally necessitate a repetition of the work, besides causing much trouble and needless nervous strain.

The bringing together of substances as directed so that chemical changes take place is in itself of little cultural value. Most of the manual occupations are quite as complex and require fully as much thought and planning. A notebook record giving the results of experiments in such terms as—dense red fumes arose, a bright flame was seen, a white precipitate formed, a pop and an explosion followed, a pungent odor was given off, and linked with a “hence” or a “therefor” evidently shows that the inwardness of the phenomena still remains practically untouched.

“A primrose by the river’s brim  
A yellow primrose was to him,  
And nothing more.”

The exercises call for reflection and require prolonged concentration of attention. What is the nature of the changes presented in the experiment? Write equations for the actions. What underlying principle is illustrated? Give your interpretation of the phenomena. Is the experiment related to some previous one or to a class-room illustration? Point out how your results differ from the statements of the text. How do you account for such discrepancies? Do not stop with knowing, but keep on doing until the knowing becomes a habit ever yours.

"Den schlechten Mann muss man verachten,  
Der nie bedacht, was er vollbringt.  
Das ist's ja, was den Menschen zieret,  
Und dazu ward ihm der Verstand,  
Das er im innern Herzen spüret,  
Was er erschafft mit seiner Hand."

### 23. Preparation of the Hydrogen Halides.

a. Place small quantities of each of the following substances in separate test tubes: Fluorspar, common salt, sodium or potassium bromide, sodium or potassium iodide. Add several drops of concentrated sulphuric acid to each. Describe what happens in each case. Blow moist air across the mouth of the tubes. (?) Hold a piece of blue litmus paper near the mouth of each tube. (?) Hold a strip of filter paper dipped in starch paste in the mouth of each tube. (?) Bring a rod dipped in ammonium hydroxide near the mouth of each test tube and gradually lower it into the tube. (?) Try the effect of heating if the action in the cold is slight. (?) What evidence is there that the gases evolved in some of the interactions are complex? In the reactions where you suspect a mixture of gaseous products try to identify some of those not yet recognized. Remove the residues from the tubes, and examine the tubes. Do any of them show signs of corrosion. (?)

b. Perform another series of experiments using the same salts, but substitute concentrated phosphoric acid for sulphuric. Compare the results with those noted in the trials with sulphuric acid.

c. Arrange the halogen hydrides in the order of their stability, and compare this order with their heats of formation. (K. 284.)

What facts gathered here support the statement that hydrogen iodide is a reducing agent? (K. 116; S. 167.)

Beginning with this chapter include in your record equations for the interactions studied.

Which of the foregoing methods of preparation is evidently not suited in obtaining the halogen hydride in a fairly pure state?



d. In separate test tubes place about 1 g. of each of the following chlorides: Lead chloride, prepared in Exp. 18, ammonium chloride, mercuric chloride, and calcium chloride. Add a few cc. of concentrated sulphuric acid to each chloride, and test for the presence of hydrogen chloride at the mouth of the test tube by one of the methods employed in a. Heat the contents of the tubes if no action occurs at room temperatures. (?)

#### 24. Preparation of the Halogens.

a. In separate test tube place about a gram of each of the following substances:—Manganese dioxide, cupric oxide, lead dioxide, litharge, potassium chlorate, potassium permanganate. Prepare some test papers, by dipping strips of filter paper in starch paste to which you have added 2-3 drops of potassium iodide solution.

Add a few drops of concentrated hydrochloric acid to the first test tube, and note the color (?) and odor (?) of the gas. If no action takes place in the cold, heat gently. Dip into the gas one of the test papers. (?) Suspend one of the test papers in the gas for a minute or two. (?) Empty the contents of the tube or set it aside; then treat the contents of the second tube with hydrochloric acid and test the gas as before. (?) Experiment with the other substances in the same way, and note carefully differences in behavior. How do you account for the differences? Do not heat the tube containing the potassium permanganate.

b. Powder about 1 g. of sodium chloride and mix it with about 2 g. of pulverized manganese dioxide. Place the mixture in a test tube, and add 2-3 cc. of sulphuric acid (1 Acid : 1 Aq.) In mixing sulphuric acid and water add the acid cautiously to the water. Stir up the contents of the test tube, and heat gently, and identify the gas. (?) What other substances might be substituted for the common salt in this experiment? (Refer to Exp. 23d.) For the manganese dioxide. (?)

Modify the above experiment by using potassium bromide in place of sodium chloride. (?)

Finally try the experiment using potassium iodide instead of potassium bromide. (?)

**24 A. Properties of Iodine and Bromine.** Place 20 cc. of water in each of two test tubes. Add a drop of bromine to one test tube and a few crystals of iodine to the other. Shake the tubes to hasten solution. Care:—Do not spill bromine on the hands. Iodine stains may be removed by washing with hypo, a solution of sodium thiosulphate.

Divide the solution of each tube between four test tubes. Add 2 cc. of ether to one of the samples of bromine water, and to one of the iodine solutions. After shaking the tubes note the relative solubility in water and ether as shown by the depth of color of the layers.

To another pair of solutions add 2 cc. of carbon disulphide and shake. (?)

To the third pair add chloroform. (?)

To the fourth pair add 10-15 cc. of starch paste. (?)

To a few crystals of iodine add 5 cc. of water and shake. Now add a small crystal of potassium iodide and shake again. (?) Interpret.

What is tincture of iodine?

**25. Preparation of Hydrobromic Acid.** (Optional; two students working together.)

Set up the apparatus figured on p. 108 K. or S. p. 162. In the flask, 250 cc. capacity, place about 5 g. of red phosphorus mixed with an equal volume of sand (use of this?) and 5 cc. of water. In the U-tube place broken glass mixed with a little red phosphorus (use of this?). Pour into the drop funnel 10 cc. of bromine, using a funnel in transferring the bromine to the drop funnel. Extreme care should be used in handling bromine. To start the reaction, allow the bromine to trickle slowly on the phosphorus. Collect the gas by dissolving it in water so arranged that the solution can not suck back into the connecting U-tube and generator.

Try the effect of moist air on the issuing gas. (?) Hold in the gas a glass rod dipped in ammonium hydroxide. (?) Test its action on moist litmus paper. (?)

Test the action of a portion of the acid on zinc. (?) Mix another portion with manganese dioxide and heat gently. (?) Add a few drops of chlorine water to another portion. (?) Taste a drop of the diluted solution. To another portion



add a few drops of silver nitrate solution, and expose the precipitate to sunlight. (?)

If water were allowed to trickle from the drop funnel of the apparatus what substance would be suitable to have in the flask instead of the phosphorus and sand mixture for the preparation of hydrogen bromide?

What advantage has this method of preparing hydrobromic acid over the interaction of sulphuric acid and a bromide? (Cf. Exp. 23a.)

**26. Preparation of Hydriodic Acid.** (Hood; Optional; Two students working together.)

Place 10 g. of powdered iodine in a flask containing about 75 cc. of water, and provided with a one-hole cork and short glass tube. Pass hydrogen sulphide into the mixture of iodine and water. Use the same form of generator as was used in preparing hydrogen. For preparing hydrogen sulphide use ferrous sulphide in place of zinc. At first the cork on the flask containing the iodine and water must be loosened occasionally to allow the air to be displaced by the hydrogen sulphide. Shake the iodine and water mixture constantly to hasten the process. Describe what happens. Continue passing the gas until the solution no longer becomes brown on shaking. Warm and filter the solution. What is the residue on the filter?

Distil the filtrate fractionally, figure 9, collecting first the portion that comes over between 99 and 100, and then the

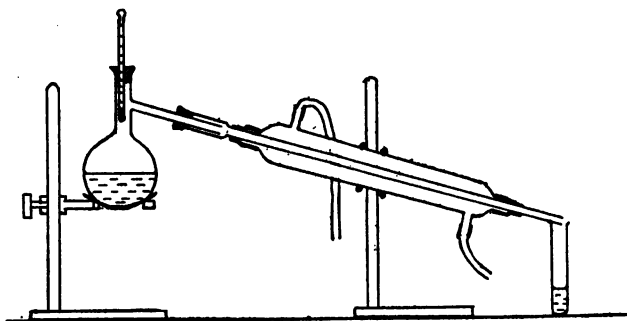


Fig. 9.

fractions boiling between 100-105, 105-110, and so on. Use a small flame and place a gauze under the distilling flask. Stop when the liquid is nearly all distilled off. Note the highest temperature reached. Pour the liquid left in the flask into a test tube and keep the series of distillates for the following experiments:

Add silver nitrate solution to the different fractions, using only a part of the solution in the case of the two highest boiling fractions. At what temperature did the most concentrated acid distil over?

Try the action of a portion of the strong acid on zinc. (?) Test with litmus paper the higher boiling fractions. (?) Warm some of the concentrated acid with a little powdered manganese dioxide?

What substance causes the color of the higher fractions and of the residue? Test your conclusion by an experiment.

Record the maximum boiling temperatures of aqueous solutions of hydrogen chloride, hydrogen bromide and hydrogen iodide and the corresponding concentrations of these mixtures. (K. 50, 108, 115; S. 120, 163, 167.)

#### 27. Properties of Chlorine. (Hood.)

a. Fit up a 250 cc. flask with a drop funnel and delivery tube as shown in Fig. 10. Since chlorine gas destroys rubber

tubing make the rubber connections short. Test the apparatus to see that it is air-tight. Place in the flask about 20 g. of potassium permanganate, and from the drop funnel admit drop by drop hydrochloric acid (1:1). Regulate the flow so that too rapid generation of gas is not produced. When the air has all been displaced from the gene-

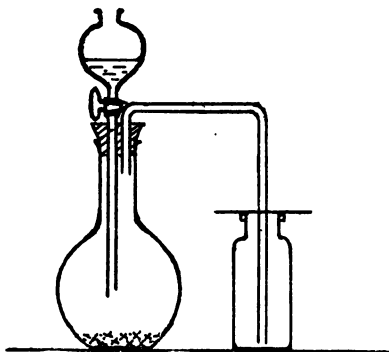


Fig. 10.

rator (how judge?) fill four dry, wide-mouth bottles with the gas. Cover the bottles with glass plates as soon as they are full.

b. When all the bottles have been filled pass a few bubbles of gas into 5 cc. of solutions of potassium bromide and potassium iodide in separate test tubes. (?) Add a few drops of carbon disulphide to each test tube and shake. (?) Interpret the results. Pass the superfluous gas into a solution of sodium hydroxide.

Dismantle the chlorine generator when sufficient gas has been collected, pour the contents of the generator into the sink and flush with water.

c. Fill a wide-mouth bottle with hydrogen and place it mouth to mouth with a bottle of chlorine. Mix the gases by inverting a few times but do not conduct this operation in direct sunlight. Hold the mouth of each bottle near a Bunsen flame. (?) Blow the breath into one of the bottles (?), and to the other add a few drops of water; shake, and test the solution with litmus paper. (?)

d. In one of the bottles of gas place moist red and blue litmus paper; a paper on which is ordinary writing and also printing; a few blades of grass, and a piece of colored calico. (?)

e. In another bottle of gas scatter a pinch of finely powdered antimony. (?) (K. 325; S. 468.)

f. Connect a glass nozzle with the illuminating gas supply, and lower a small, burning gas flame into the fourth bottle. (?) Blow the breath gently into the bottle after withdrawing the jet. Interpret the results.

**Identification of Halogen Compounds.** If you were given four white substances and told that they were the fluoride, chloride, bromide, and iodide of some metal, state what experiments you would make in order to identify the halogen constituent of each. Negative results, showing that one is not a chloride, bromide, or iodide, and is therefore a fluoride, must be confirmed by an experimental test.

Ask the instructor for an unknown halogen compound and then proceed to identify it, recording the different tests that you conducted. Report your conclusion to the instructor.

## CHAPTER VIII.

### ACIDS, BASES, SALTS. CHEMICAL EQUILIBRIUM.

#### 28. Properties of Acids and Bases.

a. Prepare a sample of dilute hydrochloric acid by adding 5 cc. of the concentrated acid to 25 cc. of distilled water, and examine the solution in respect to taste, behavior toward litmus, phenolphthalein (use but 1 or 2 drops), action on a bit of magnesium ribbon, action on baking soda or marble. Add one drop of concentrated acid to 10 cc. of water and see whether the solution tastes sour. Determine the dilution at which you can barely detect the sour taste of the acid. (?) Test the very dilute solution with litmus. (?)

Test a sample of dilute nitric acid (1: 10) with the same reagents used in determining the characteristics of hydrochloric acid. (?)

The above properties are shown by other acids.

What element (or elements) is found in the formulas of all the acids on the laboratory shelf of liquids? If HA is adopted as the general formula for acids, underscore the radical represented by A in the following acids,—sulphuric, carbonic, nitric, chloric, hydriodic, perchloric, sulphurous, phosphoric. Indicate the valence of the radicals underscored.

b. Dissolve 5 g. of lye in 50 cc. of water, and test the solution with the same reagents employed in a. Repeat the experiments with a sample of lime water. Write the chemical name and formula for,—lime water, lye, caustic potash. Other bases have the properties common to caustic soda. What radical is common to bases? Write a general formula for bases.

Tabulate the observations in a and b under the following heads:—Taste, litmus test, phenolphthalein test, action on magnesium, action on baking soda.

c. In separate test tubes, or watch glasses, place about 1 cc. of the following solutions and note their behavior toward litmus:—borax, ferric chloride, magnesium sulphate, sodium

acid carbonate, sodium carbonate, sodium acid sulphate, sodium chloride. (?) How do you account for the differences in behavior of the solutions toward litmus? ( K. 130; S. 163, 353.)

**29. Neutralization.** (Two students working together.)

a. Mount a pair of clean burettes fitted with nozzles. (Exp. 4.) Prepare about 200 cc. of sodium hydroxide solution by dissolving 10 g. of the solid. Prepare also about 100 cc. of dilute hydrochloric acid by adding 10 cc. of concentrated acid to 100 cc. of water. Fill one of the burettes with base and the other with acid. Now, into a small beaker or flask run 10 cc. of the acid and add two drops of phenolphthalein. Place the vessel under the other burette and allow the alkali to run into the acid slowly, stirring constantly, until a drop produces a perceptible pink tint in the whole solution. If too much alkali is accidentally added, run in a drop or two of acid to discharge the pink color, and then add alkali again until the solution becomes faintly pink. Finally read the volumes of acid and base used. Make a second and third trial with fresh quantities of acid using 15-20 cc. in each determination. Read carefully the volumes of acid and base used, and calculate the ratio of the volumes in each titration. Arrange your data in the form of a table under the following heads:—Determination number, volume of alkali, cc., volume of acid, cc., indicator, ratio of base to acid. Interpret the ratios.

Find the average of your ratios and express the percentage difference between the mean value and the single determination differing most from it. Compute the volume of alkali that would be required to neutralize 25 cc. of your acid.

b. (Quant.) Neutralize exactly 25 cc. of the acid with your solution of alkali, and note the volume of alkali required. Transfer the solution to a weighed evaporating dish, rinse the vessel, and add the rinsings to the main solution. Evaporate to dryness on the water bath, and finally heat the residue gently over a small Bunsen flame. Allow it to cool and then weigh. What is the residue?

Assuming the equation for the neutralization (K. 122; S. 241), calculate from the weight of the residue the number of grams of hydrogen chloride and sodium hydroxide per liter of your solutions. Express the concentration (strength) of your solutions in terms of normal.

c. (Quant.) Determine the concentration of your alkali solution by titration against the standard sulphuric acid in the laboratory. Make at least two determinations, and do not use less than 10 cc. of acid for each. Express the concentration of your alkali in terms of normal. Compare this value with that obtained in b, and express the percentage difference. Submit your result to the instructor for criticism.

d. (Quant.) Secure from the instructor a sample of an acid, an unknown, and determine its concentration by titration against your standard alkali, and report the value to the instructor.

What weight of ammonium chloride will result from the neutralization of 50 cc. of fifth normal ammonium hydroxide? What volume of fourth normal ( $\frac{N}{4}$ ) acid will be required?

### 30. Double Decomposition. Precipitation.

a. Place solutions of hydrochloric acid (dil.), and potassium chlorate, potassium chloride, and some other chloride in as many test tubes, and add a few drops of silver nitrate solution to each. (?) (K. 134-8; S. 187.) Bearing in mind that salts in solution generally interact in such a way that an exchange of radicals takes place, construct equations for the actions. How can you tell whether the precipitate is silver chloride or a nitrate, or both? Which is it? (Appendix 8.) Do all substances containing chlorine give silver chloride by double decomposition in solution? What radical must a substance contain to yield silver chloride by interaction with silver nitrate solution?

By referring to the table of solubilities (Appendix 8) try to predict which of the following solutions will give precipitates by the addition of silver nitrate solution,—potassium iodide, potassium bromide, potassium fluoride. Give the reasoning upon which your prediction is based. Confirm your supposition by suitable experiments. (?)

Suggest some other nitrate that you think would yield a precipitate of a chloride from the solutions of chlorides used above and verify your answer.

b. Filter off one of the precipitates of silver chloride obtained and add a few drops of silver nitrate to the filtrate. (?) If no precipitate forms what inference do you draw? If a precipitate forms formulate your ideas of how you would proceed by this method to obtain a filtrate free from chloride.

c. Add a few drops of silver nitrate solution to solutions of sodium carbonate and sodium phosphate in separate test tubes. (?) Add nitric acid to the contents of the tubes until no further change occurs. (?) Also add a few drops of nitric acid to one of the precipitates of silver chloride. (?) How would the presence of dilute nitric acid in solutions of sodium phosphate and potassium chloride affect the interaction of the solutions with silver nitrate?

Add a few drops of ammonium hydroxide to silver chloride. (?)

Formulate a test for a soluble chloride that will distinguish it from a phosphate or carbonate. Would this test also distinguish the chloride solution from the solution of an iodide or bromide?

Ascertain whether the tap water contains chlorides. (?)

d. To a few drops of silver nitrate solution add some ammonium hydroxide and so obtain a solution of ammonio-silver nitrate. Now add a solution of sodium or potassium chloride. (?) Is the silver radical present? Which compounds alone give silver chloride by double decomposition?

e. To solutions of sodium carbonate, sodium phosphate, and sodium sulphate in separate test tubes add a few drops of barium chloride solution. Assuming that an exchange of radicals occurs, write equations for the actions, and determine by referring to the table of solubilities, which of the products is the precipitate. Add a few drops of barium chloride solution to samples of dilute sulphuric and phosphoric acids. (?) Add a little hydrochloric acid to each. (?) Interpret the results. (?) If solutions of sodium phosphate and sodium sulphate are acidified with hydrochloric acid,

would you expect precipitates to form in either of them upon the addition of barium chloride? Confirm your answer by experiments. (?)

Formulate a test for the sulphate radical. Will your test enable you to distinguish sulphate solutions from a carbonate solution? From a solution of a phosphate?

Suggest an experimental method of distinguishing between solutions of carbonates and phosphates.

When a solution of ammonium molybdate is added to a solution of a phosphate, e. g., sodium phosphate, to which a few drops of nitric acid have been added, a yellow precipitate is obtained either at once or upon warming the solution. In dilute solutions the precipitate only appears after the solutions have stood for a time. Try the molybdate test for the phosphate radical. (?) The formula of the precipitate is complex. Do not attempt an equation for the reaction. (K. 491; S. 483.)

f. Suppose that you were given four white substances and that you knew them to be a carbonate, chloride, phosphate, and sulphate of a metal. State what tests you would make, and what reasoning you would use, in order to identify the acid radical in each. Ask the instructor for a specimen of one or more of these salts and proceed to identify it. Record the experiments that you conduct with the unknown and give the reasoning you use in identifying the substance.

### 31. Examples of Reversible Reactions.

To a concentrated solution of sodium hydrogen sulphate add about an equal volume of concentrated hydrochloric acid. (?) Filter off the precipitate and dry it by pressing it between filter paper. Dissolve it in water and then evaporate the solution to dryness on the water bath. Describe and name the product.

Write the equation for the action. What relation does this action bear to that in Exp. 23d? What factors determine the direction of a reversible reaction? (K. 134-8; S. 174-9.)

Recall the reduction of ferric oxide by means of hydrogen and the action of steam on heated iron. Specify the conditions that must be observed to complete the reaction in either direction.



## CHAPTER IX.

### AMMONIA AND NITRIC ACID.

#### 32. Ammonia and Ammonium Compounds.

a. Fit a small flask with a one-hole stopper and L-tube, and connect the latter with a U-tube. Put some water in the latter, filling it to such a height that air can return to the flask without drawing back water. Test the apparatus to see that it is air-tight. Prepare a mixture of powdered quick-lime and sal ammoniac, about 10 g. of each. Has the mixture an odor? Place it in the flask and warm gently. After the air has been displaced the whole of the gas should dissolve in the water. Name the solution thus prepared.

b. Hold a glass rod dipped in concentrated hydrochloric acid over the solution. (?) Boil about 5 cc. of the solution in a test tube and note the odor from time to time. (?) Compare this behavior of the solution with that of hydriodic acid. (Exp. 26.)

c. Neutralize the remainder of the ammonium hydroxide prepared with sulphuric acid and evaporate the solution to dryness on a water bath. (?) Scrape the residue into the middle of the dish, invert over it a small funnel the stem of which has been plugged with paper, and heat the dish strongly for some time. Is the sublimate identical with the residue? For answer test it for the sulphate radical and the ammonium radical. The test for the latter may be conducted as in a, or by adding 2-3 cc. of sodium hydroxide solution to a solution of the sublimate and then gently warming. Note the odor. (?)

d. (Two students working together.) In the middle of a hard glass tube pack a plug of asbestos fiber about 0.5 cm. thick, and place about a gram of ammonium chloride on one side of the plug. Mount the tube in a horizontal position and place strips of moist filter paper in both open ends of the

fractions boiling between 100-105, 105-110, and so on. Use a small flame and place a gauze under the distilling flask. Stop when the liquid is nearly all distilled off. Note the highest temperature reached. Pour the liquid left in the flask into a test tube and keep the series of distillates for the following experiments:

Add silver nitrate solution to the different fractions, using only a part of the solution in the case of the two highest boiling fractions. At what temperature did the most concentrated acid distil over?

Try the action of a portion of the strong acid on zinc. (?) Test with litmus paper the higher boiling fractions. (?) Warm some of the concentrated acid with a little powdered manganese dioxide?

What substance causes the color of the higher fractions and of the residue? Test your conclusion by an experiment.

Record the maximum boiling temperatures of aqueous solutions of hydrogen chloride, hydrogen bromide and hydrogen iodide and the corresponding concentrations of these mixtures. (K. 50, 108, 115; S. 120, 163, 167.)

#### 27. Properties of Chlorine. (Hood.)

a. Fit up a 250 cc. flask with a drop funnel and delivery tube as shown in Fig. 10. Since chlorine gas destroys rubber

tubing make the rubber connections short. Test the apparatus to see that it is air-tight. Place in the flask about 20 g. of potassium permanganate, and from the drop funnel admit drop by drop hydrochloric acid (1:1). Regulate the flow so that too rapid generation of gas is not produced. When the air has all been displaced from the gene-

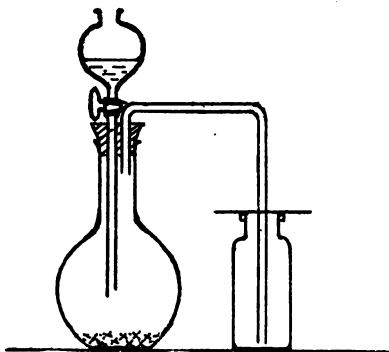


Fig. 10.

rator (how judge?) fill four dry, wide-mouth bottles with the gas. Cover the bottles with glass plates as soon as they are full.

b. When all the bottles have been filled pass a few bubbles of gas into 5 cc. of solutions of potassium bromide and potassium iodide in separate test tubes. (?) Add a few drops of carbon disulphide to each test tube and shake. (?) Interpret the results. Pass the superfluous gas into a solution of sodium hydroxide.

Dismantle the chlorine generator when sufficient gas has been collected, pour the contents of the generator into the sink and flush with water.

c. Fill a wide-mouth bottle with hydrogen and place it mouth to mouth with a bottle of chlorine. Mix the gases by inverting a few times but do not conduct this operation in direct sunlight. Hold the mouth of each bottle near a Bunsen flame. (?) Blow the breath into one of the bottles (?), and to the other add a few drops of water; shake, and test the solution with litmus paper. (?)

d. In one of the bottles of gas place moist red and blue litmus paper; a paper on which is ordinary writing and also printing; a few blades of grass, and a piece of colored calico. (?)

e. In another bottle of gas scatter a pinch of finely powdered antimony. (?) (K. 325; S. 468.)

f. Connect a glass nozzle with the illuminating gas supply, and lower a small, burning gas flame into the fourth bottle. (?) Blow the breath gently into the bottle after withdrawing the jet. Interpret the results.

**Identification of Halogen Compounds.** If you were given four white substances and told that they were the fluoride, chloride, bromide, and iodide of some metal, state what experiments you would make in order to identify the halogen constituent of each. Negative results, showing that one is not a chloride, bromide, or iodide, and is therefore a fluoride, must be confirmed by an experimental test.

Ask the instructor for an unknown halogen compound and then proceed to identify it, recording the different tests that you conducted. Report your conclusion to the instructor.

**34. Reduction of Nitric Acid to Ammonia. (Optional.)**

a. Dissolve a small crystal of sodium nitrate in 2 cc. of water. Add about the same volume of concentrated sodium hydroxide solution and some aluminum turnings or chips. Heat gently and test the gas evolved with litmus paper. (?) Note the odor of the gas. (?) Is it inflammable? (K. 16, 87; S. 68, 302.)

b. To 5 g. of zinc in a test tube add dilute sulphuric acid, and when the evolution of hydrogen is in progress add concentrated nitric acid at the rate of a drop a minute. If ammonia is formed by complete reduction of nitric acid where will it be found, and in what condition? Test for its presence. (?)

## CHAPTER X.

### HYDROGEN SULPHIDE, SULPHUR DIOXIDE, CARBON DIOXIDE.

#### 35. Hydrogen Sulphide. Preparation and Properties.

a. Set up the apparatus used in the preparation of hydrogen, but it is advisable to use a smaller flask. In the flask place about 20-30 g. of ferrous sulphide, and pour in through the thistle tube sufficient hydrochloric acid to cover the sulphide. Later if the action becomes too slow add a little concentrated acid at intervals. When the gas is not being used lower the delivery tube into a solution of sodium hydroxide. This prevents its escape into the room.

Note the odor of the gas and apply to it a strip of test paper,—filter paper dipped in lead nitrate solution. (?) When the air has been displaced from the generator, and the evolution of gas is brisk, collect several bottles of it over water. In connection with this operation what evidence, if any, of the solubility of the gas in water did you observe? Is the gas combustible? Does it support combustion? To one of the bottles of gas covered with a glass plate add a few drops of bromine water and quickly close the mouth of the bottle with the palm of the hand and shake. (?) What evidences of action did you note?

Set fire to the gas. Describe the color of the flame and note if moisture is deposited on a dry beaker held for an instant over the flame. Identify by its odor any gas formed by the burning. (?) Hold a porcelain dish in the middle of the flame for a few moments. (?) What is deposited? Is it therefore probable that this substance exists uncombined in the interior of the flame? (S. 251). Pass the gas into concentrated sulphuric acid. (?) Identify one of the products of the interaction by the odor of the gas issuing from the acid. (?) What other products do you recognize? Lead hydrogen sulphide into dilute nitric acid and try to identify some of the products.

b. Take about 10-15 cc. of water in a test tube and saturate it with hydrogen sulphide. Test the solution with litmus paper. (?) Assign a name to the solution. Pour a drop of the solution on the test paper used above. In a test tube boil 5 cc. of the solution and note the odor of the vapor from time to time, and finally try to detect the presence of the gas in the vapor by means of the test paper. Can the gas be driven off completely by boiling? Compare this behavior with ammonium hydroxide and hydrochloric acid. Expose the remainder of the solution to the air until the next laboratory period. Explain the turbidity.

c. Saturate about 10 cc. of ammonium hydroxide with the gas. Test with litmus paper. (?) To a few drops of the solution just prepared add dilute hydrochloric acid and note the odor of the escaping gas. (?) Acidify a portion of the caustic soda solution in which the superfluous gas was absorbed in the course of the experiments. (?) To the remainder of the ammonium hydrogen sulphide solution add a little powdered roll sulphur, and shake from time to time. (?) Is sulphur soluble in water? What is here to be inferred? When the solution has become very yellow in color, filter. What is the name of this solution? (K. 372; S. 371.) Acidify the filtrate with dilute hydrochloric acid. (?)

d. Take six clean test tubes and obtain 2-3 cc. of the solution of each of the following substances. Dilute each specimen with 10-20 cc. of water and saturate with hydrogen sulphide: (a) Cupric sulphate (?), (b) Lead nitrate (?), (c) Cadmium sulphate (?), (d) Zinc acetate (?), (e) Ferrous ammonium sulphate (?), (f) Potassium sulphate (?).

Pour away a part of the contents of each tube, add a large excess of dilute hydrochloric acid, and shake (?). Explain the results. Divide the metallic sulphides obtained as precipitates into two classes, and characterize those classes.

Saturate a diluted solution of ferrous ammonium sulphate which has previously been acidified with dilute hydrochloric acid with hydrogen sulphide. (?) Now add ammonium hydroxide drop by drop until the solution shows an alkaline reaction. (?) Filter off the precipitate, and lead more hydrogen sulphide into the filtrate. (?) If no further precipitate forms,

what may you infer? If a precipitate is obtained how would you proceed to obtain a filtrate free from ferrous sulphate?

e. Imagine that you have mixed solutions of cupric sulphate, ferrous sulphate and potassium sulphate. State how you would proceed in the light of the facts ascertained in d, to separate the metallic radicals from the solution. Try your plan, but before doing so submit it to the instructor for suggestions and criticism.

f. What volume of hydrogen sulphide will be produced by dissolving 20 g. of ferrous sulphide, 90 per cent pure, in dilute hydrochloric acid?

g. What volume of air is necessary to burn a liter of hydrogen sulphide?

h. Suggest a method of obtaining zinc sulphide from a solution of zinc chloride. (?) Try your method. (?)

### 36. Sulphur Dioxide. Preparation and Properties.

a. Enumerate the experiments in which you identified sulphur dioxide.

b. Place in separate test tubes specimens of sodium carbonate, sodium sulphite, and sodium thiosulphate (hypo), and add to each a little dilute hydrochloric acid. (?) Note the odor of the gaseous products. (?) How would you identify the gas evolved from the carbonate? (Exp. 9.) When the crystals have dissolved examine the contents of the test tubes and point out any difference that could be used to distinguish the sulphite from the thiosulphate.

c. Set up on a sand-bath the apparatus used in preparing hydrogen sulphide. Connect the exit tube to a washing-bottle of the type shown in Fig. 11. This bottle is to serve here as

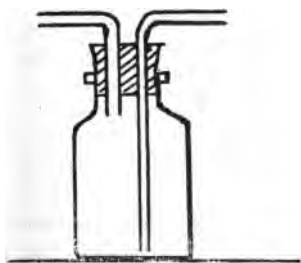


Fig. 11.

a safety trap (how and why?) and is to be left empty. The shorter tube of the washing bottle should be toward the generator. Why not the reverse? Into the flask put 10-15 g. of copper chips or turnings, and through the thistle tube pour about 25 cc. of concentrated sulphuric acid. Test the apparatus to see that

it is air-tight. Heat the flask and contents until a gas begins to evolve. Then moderate the heating so that a slow steady stream of gas will escape. While the gas is not being collected pass it into water and a solution of sodium hydroxide. Save these solutions as well as the contents of the generator.

Collect four bottles of gas by displacement of air. Invert the last bottle of gas collected over water. (?) If any gas remains in the bottle, what should you expect it to be?

Test the odor of the gas, and test its action on moist litmus paper. (?)

In the third bottle place the following substances:—Litmus paper, colored cloth, paper on which is ordinary writing and printing, green grass or a flower. Stopper the bottle and allow it to stand until the next laboratory period. (?) Then explain the changes.

Does the gas burn? Does it support combustion?

c. Test the aqueous solution of the gas with litmus. (?) Name it. Boil about 5 cc. of the solution in a test tube and note the odor from time to time. (?) Finally test the remaining liquid with litmus paper. (?) What other solutions of gases examined before behaved in the same way? Test a portion of the aqueous solution for the sulphate radical by adding barium chloride and then pure hydrochloric acid. (?) Is the action reversible? Add bromine water to another portion of the sulphurous acid until the tint is permanent, and then test for the sulphate radical. (?) What property of bromine water is shown by this experiment. (?)

Add potassium permanganate solution to another portion of the solution a few drops at a time, as long as the pink color disappears. Test the solution for the sulphate radical.

Leave the remainder of the aqueous solution in the bottle for several days exposed to the air. Then test for the sulphate radical. (?)

d. Place about 10 cc. of potassium permanganate solution in each of three test tubes. In three more place solutions of potassium dichromate. Acidify each solution with 2 cc. of concentrated sulphuric acid. Into one of the samples of permanganate solution pass sulphur dioxide or add the aqueous solution of the gas until the pink color is completely dis-



charged. Treat one of the dichromate solutions in the same way. (?) Into another pair of the solutions lead hydrogen sulphide until no further change occurs. (?) In the third set place a few pieces of granulated zinc. (?) Explain the actions. (K. 500, 490; S. 492, 478, 253.) What property of sulphurous acid and hydrogen sulphide is illustrated by these experiments? Classify all the reactions of this section under one head.

e. To a small portion of the sodium hydroxide used to absorb the superfluous gas, add dilute hydrochloric acid until the solution reacts acid toward litmus. (?) If any gas is evolved note its odor. (?) To another small portion of the solution add a little bromine water. Test both solutions for the presence of the sulphate radical. (?)

f. Describe the contents of the generator. If any copper chips remain remove them from the residue after diluting with a small volume of water, rinse them and return to the shelf bottle. Formulate a plan of obtaining crystals of blue vitriol from the residue. (?) Submit your plan to the instructor for criticism and finally show him your yield of crystals.

### 37. Carbon Dioxide. Preparation and Properties.

a. Enumerate the reactions of previous experiments in which carbon dioxide was identified. What property of carbon dioxide have you used for its identification?

b. Place a few small pieces of marble or limestone in hard glass test tube fitted with a cork and L-tube, and heat strongly. Pass the gas through 5 cc. of lime water in a test tube. (?) What is the residue? Place it in an equal bulk of water, and set aside for a few minutes. Then add about 20 cc. of water and shake vigorously and filter. Test the filtrate with litmus paper. (?) Name the filtrate.

c. Set up a generating flask of the type used in preparing hydrogen, and connect it with a wash-bottle containing some distilled water. Put in the flask pieces of marble and pour upon them dilute hydrochloric acid. Collect the gas in bottles by downward displacement of air or over water. What is the function of the wash-bottle? Verify your answer experimentally. (?) What substance could be substituted for marble in this experiment?

To one bottle of the gas add some distilled water, close with the hand, and shake. Test the solution with litmus. (?)

Has the gas odor? Does it support combustion? Pour a bottle of the gas over a lighted taper. (?)

Collect a bottle of the gas over water and keep it inverted over water until the next laboratory period. (?)

What name is sometimes applied to the aqueous solution of the gas? Boil an aqueous solution of the gas for a minute and then test the remaining liquid with lime water. (?)

Pass the superfluous gas from the generator into 50 cc. of ammonium hydroxide and store it in a corked vessel for future use.

By means of a tube blow air from the lungs through lime-water. (?) How could you determine the proportion of carbon dioxide in the air expelled from the lungs?

Explain the statement,—The carbon dioxide present in the water was fixed by adding a few cc. of strong caustic potash.

d. In a flask place about 100 cc. of lime water and pass a stream of carbon dioxide persistently through the solution. (?) Divide the clear liquid into three parts. (If not quite clear, filter it). Boil one portion gently in a small flask fitted with a delivery tube and pass the vapors into lime water. (?) What is the solid in the flask? Explain temporary hardness as applied to waters. To the second portion add lime water. (?)

To the remainder of the temporary hard water add 1 cc. of soap solution and shake. Does a lather remain? Is a precipitate present in the liquid? Save the sample.

Add a few drops of soap solution to approximately the same volume of distilled water, and shake. Does a lather form? If not, continue adding soap solution, a few drops at a time, until a lather remains on the liquid for a minute. Determine the volume of soap solution required to form a lather in the temporary hard water. What is the precipitate that forms in the temporary hard water?

Summarize the methods of softening temporary hard water.

Dissolve a small crystal of magnesium sulphate in a test tube full of distilled water. To another sample of water add a pinch of powdered gypsum; shake persistently, and then

filter. You now have two samples of permanent hard water. Add a few drops of soap solution to each and determine the volume of soap solution required to form a lather. What is the precipitate that forms? (K. 253; S. 335.)

Give a method of softening permanent hard water that does not involve the use of soap. (S. 392.)

What is the commercial name of crystallized magnesium sulphate?

**38. Sodium Carbonate by the Solvay Process.** (Two students working together. Optional.)

Unite the solutions that were obtained by passing the waste carbon dioxide of the previous experiment into ammonium hydroxide, and saturate the solution with sodium chloride by persistently shaking in a corked flask. Decant the clear liquid into another bottle, fitted with two tubes, one of which reaches to the bottom. If because of delay a precipitate has appeared, proceed without decanting the solution. Through the longer tube pass in carbon dioxide until the solution is saturated. This operation will require at least half an hour, and generally longer. During the absorption of carbon dioxide the exit tube should be closed to prevent waste of the gas. Close the tubes with caps of rubber tubing plugged with glass rods and set aside over night. (?) Filter off the deposit and dry by pressing between filter papers.

Dissolve in water a little of the solid, and test the reaction of the solution with litmus. (?) If the solution is not acid explain why it is not so.

Add any dilute mineral acid to a part of the solid. (?)

Heat the rest in a test tube fitted with a cork and delivery tube, and pass the gas into lime water. When gas ceases to be given off, dissolve the cold residue in a very little water, test the reaction of the solution with litmus paper, and set it aside to crystallize in an open, shallow dish. (?) Explain the reaction with litmus. (?) Dry the crystals and try the effect of acid upon them. (?)

What are the commercial names of sodium hydrogen carbonate and crystallized sodium carbonate?

**39. Identification of Unknown Salts.**

a. Make a list of the negative radicals studied in the laboratory experiments, and place opposite each the gases, if any, that would be evolved by the addition (a) of dilute hydrochloric acid, and (b) a little concentrated sulphuric acid and warming, to soluble salts of the radicals. Arrange your data in tabular form.

b. Apply to the instructor for two unknown substances, and ascertain by means of experiments the acid radical in each. Report your conclusion to the instructor with a report giving the reasoning used in identifying the unknowns.

## CHAPTER XI.

### THE ATMOSPHERE, FLAME. OXIDATION AND REDUCTION.

#### 40. Components of Air.

a. Place 3-5 cc. of clear barium hydroxide solution in the bottom of a small beaker and leave it exposed to the air until the end of the laboratory period. (?) Relate this experiment to the observations made when air from the lungs was blown through lime water. (Exp. 37 c.)

b. (Quant.) On watch glasses expose to the air weighed specimens of blue vitriol, fused calcium chloride, Glauber's salt, and metallic sodium. Toward the close of the laboratory period weigh again, and make another set of weighings during the following laboratory period. Construct a table showing the changes in weight of the substances. Account for the observed variations in weight and write equations indicating the changes that have occurred in the different substances.

**Note 9.** When you are requested to give an interpretation of results or furnish an explanation, please bear in mind that an equation alone is not a sufficient answer.

Add a few drops of acid to the material that resulted from the exposure of sodium to the air. (?)

Explain the terms, deliquescent, efflorescent, hygroscopic.

c. Place about 5 g. of iron filings, moistened with a few drops of water in the bowl of a retort. Clamp it vertically on a retort stand so that the neck dips about 6 cm. below the surface of some water in a beaker of about 300 cc. capacity. With a file mark where the water stands in the neck of the retort. Put the stopper in place and cover it with a little melted paraffine to insure the apparatus being air-tight. Set aside until the next laboratory period, but mark the position of the water in the neck of the retort at the close of the laboratory period. Read the barometer and note the room temperature. Upon resuming the experiment mark the position of the water with a file. Read the barometer, and take the

room temperature. Insert a cork in the end of the neck of the retort in order to prevent the water from running out. Test the gas left in the bowl by means of a burning splinter of wood. Is it air? Why is the volume different from the original air? To what extent do the changes in pressure and temperature account for the observed changes in volume? Has the iron changed in any way? What does the contraction represent? Measure the volume which represents the contraction and also that of the gas that remains. Find the percentage of each of the original volume.

The experiment may be conducted with a glass cylinder in place of the retort.

d. Place a wad of copper turnings in the center of a hard glass tube. Fit the tube with one-hole corks provided with short glass tubes, and connect with the short tube of the aspirator used in determining the weight of a liter of oxygen. Fill completely with water the bottle and long tube of the aspirator and close the latter with a screw-clip. Now heat the copper red-hot and operate the aspirator so that a slow current of air will pass over the heated copper. What changes does the copper undergo. When about one-half of the water in the aspirator has run out open the cork and ascertain whether the gas in the bottle supports combustion. (?) Examine the turnings, and by bending, twisting, and scraping remove most of the scale. Compare it with the oxides of copper on the side-shelf. (?) Place the scale in the bottom of a test tube, cover it with a thin layer of powdered charcoal, and heat gently. When cold pour the contents of the tube into a small beaker and separate the components of the mixture by letting a small stream of water flow into the beaker. What is the residue in the beaker? How was copper oxide reduced in an earlier experiment?

#### 41. Bunsen Flame.

a. Prepare a clean, bright copper wire and explore the non-luminous flame with it. Watch the play of colors on the wire and explain. Cut off the gas supply until the flame is about 6 cm. high, and close the air holes until a luminous point appears at the apex of the inner cone. Heat portions of the wire coated with oxide in the luminous region of this flame.

Before withdrawing the wire lower it into the inner cone of unburnt gas to cool. (?)

b. Make a borax bead by dipping the glowing end of the platinum wire in a bit of borax on a watch glass, and then heating in the flame. Observe the behavior of the borax and explain. (K. 302; S. 350.) Bring the hot borax bead in contact with a minute particle of cupric oxide and heat it in the oxidizing region of the flame until the particle has dissolved. What is the color of the bead when cold? Now adjust the flame for reducing and heat the bead persistently in the luminous apex. (?) Explain. Finally reheat the bead in the oxidizing flame. (?)

Remove the bead from the wire by fusing it again, and giving the wire a short, sharp jerk, or by plunging the molten bead into a little dilute hydrochloric acid. Prepare a fresh bead, (it should be clear and transparent, when cool) and dissolve in it a speck of manganese dioxide. If, after heating in the oxidizing flame the bead is black, too much of the dioxide has been used. Throw the molten bead off and try again. Heat in the reducing flame and finally again in the oxidizing flame. (?) Explain.

Try the behavior of a cobalt compound with borax in the oxidizing and reducing flames. (?)

What is the source of the oxygen in these experiments?

c. Pulverize a few particles of potassium sulphate. Adjust the Bunsen flame for reducing purposes. Heat the platinum wire, touch the salt with it, and heat the adhering powder steadily in the luminous apex. After a minute or two withdraw the bead, place it upon a clean silver coin and moisten with a drop of water. (?) Explain the result. (S. 420.) What are the reducing agents here.

#### 42. Destructive Distillation.

a. Arrange the apparatus shown in Fig. 12. Fill the hard glass test tube about half full of sawdust. Heat gently at first and then strongly until no further change occurs. (?) While heating bring a flame to the end of the jet. (?) Note the odor of the gas, and its reaction toward moist litmus paper. What is the residue? Reserve it. Test the liquid in the bottle with litmus paper. (?)

b. Clean the apparatus or replace it with a fresh set and repeat the experiment using bituminous coal. (?) Save the residue.

c. Repeat the experiment using gelatine. (?)

d. Recall the heating of cane sugar. (Exps. 8, 13 a.)  
Was the experiment an example of destructive distillation?

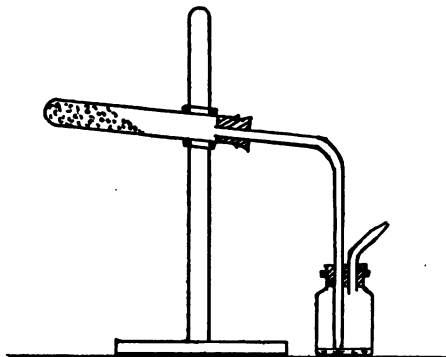


Fig. 12.

f. (Quant.) Weigh a covered porcelain crucible, and place in it about 2 g. of powdered coal, and weigh again. Place the crucible on a pipe stem triangle and heat it strongly in a flame about 20 cm. high. Make sure that the lid fits closely on the rim of the crucible. Continue the heating for five minutes after the gases no longer burn between the crucible and lid. Cool, weigh, and from the loss in weight compute the percentage of volatile matter.

Weigh out a second sample of the powdered coal, about 1 g., and heat it with the cover on until the volatile matter is gone. Then remove the lid and heat strongly, inclining the crucible slightly until constant weight is obtained. From the data now in hand compute the percentage of fixed carbon and ash.

Suggest a method of estimating the moisture in a sample of coal.

Did you note any evidence that would show that sulphur compounds are present in the sample of coal examined. What is meant by the "volatile combustible" matter of coal?



Compare your percentages with the values obtained by two other students who made determinations on the same sample of coal, and discuss jointly and with the instructor the factors responsible for the differences in values. (?)

**43. Reductions and Oxidations.**

a. Mix intimately in the mortar 5 g. of finely pulverized potassium nitrate with 2 g. of the charcoal or coke obtained in the previous experiment. Drop portions of the mixture in a red-hot crucible. (?) What gases are evolved? Test the residue with an acid. (?)

b. Mix on paper intimately (care) 5 g. of powdered potassium nitrate and 2 g. of flowers of sulphur. Throw the mixture in small portions into a red-hot crucible. (?) What gases are evolved? Dissolve the residue in distilled water and add a few cc. of pure dilute hydrochloric acid and then barium chloride solution. (?)

Write the equation for the explosion of gunpowder (S. 366; K. 349.) What product is the white smoke?

c. Mix a pinch of powdered arsenic trioxide with pulverized charcoal prepared in the previous experiment. Heat the mixture strongly in a dry test tube. (?)

d. (Optional.) Mix thoroughly equal bulks of finely powdered marble and magnesium powder. Fill a test tube to a depth of 5 cm. with the mixture, clamp in an inclined position, and heat the top layer in the Bunsen flame until the reaction begins. Apply a light to the gases escaping while the reaction is in progress. (?) If the reaction does not go to completion by itself, heat the contents of the tube again, but be careful to point the tube away from the face during the heating. Allow the test tube to cool, add a little water, and transfer the material to a small beaker and boil. If any gas is liberated upon the addition of water, note its odor and see whether it burns. Acidify the mixture with hydrochloric acid (?) and when all action has ceased, filter and wash the residue with water. Dry it on the steam-bath and prove that it is carbon. (?) What is the reducing agent in this action? Why was the hydrochloric acid added? Consider carefully the original reaction with a view of establishing what gas burned at the mouth of the tube, as the glow spread through the mixture. (?)

Evaporate the residue to dryness on a sand-bath in an evaporating dish, and finally heat whilst stirring over the free flame. Expose the product to the air until the following laboratory period. (?) What compounds previously examined behaved similarly. Interpret the result.

**f. Preparation of Amorphous Silicon. (Optional.)**

Prepare an intimate mixture of clean, finely powdered sand and magnesium powder, using equal weights of the substances, and about 5 g. of each. Fill a test tube to a depth of 6 cm. with the mixture and heat in the Bunsen flame as directed in the previous experiment. (?) Digest the residue with dilute hydrochloric acid, and apply a lighted match to the gaseous products. (?) Is there any evidence that silicon hydride is present in the escaping gas? After the action has ceased, filter and wash the residue with water. Dry it by warming, and divide into two parts. Heat one portion strongly on an inverted crucible cover. (?) Place the other portion in a test tube and digest it with caustic potash solution. (?)

**44. Alcohol by Fermentation. (Optional; two students working together.)**

Dissolve 20 g. of glucose syrup or molasses in 150 cc. of water. Break up a half yeast cake in luke warm water until it is thoroughly mashed. Add the yeast to the syrup solution, fill a flask to the base of the neck with the mixture, and fix a plug of cotton in its neck. Set the whole aside in a warm place for 3-4 days. Warm the solution and test the gas that is given off for carbon dioxide. (?)

Filter the liquid and place it in a larger flask, fitted with a cork and exit tube connected to a condenser (K. 37; S. 26.) Distill off about 50 cc., and note the odor of the distillate. (?) Place the distillate in a distilling flask fitted with a thermometer, (Exp. 26), boil with a small flame and catch the fraction that passes over between 75-93°.

Again note the odor of the distillate. (?) Test it with litmus paper. (?) Use a few drops to see whether it will burn. To the rest add a crystal of iodine and just enough sodium hydroxide solution to dissolve it. Heat gently and then cool. (?) The yellow crystalline substance that forms is iodoform, and this is the iodoform test for alcohol. Note the odor of the iodoform.

## CHAPTER XII.

### IONIZATION.

#### 45. Ionic Materials.

- a. Secure from the store-room an electrolytic cell of the type shown in Fig. 13.

Set it up on the table equipped for electrolytic experiments, by connecting in series between the terminals of a 110 volt direct current, an 8 or 16 candle power incandescent lamp and the cell wires. If the circuit is not provided with a simple switch, a single pole, a single throw knife switch should be placed in the circuit. Draw a diagram of the connections as you have made them. Bring from the side-shelf in test tubes the following materials to be

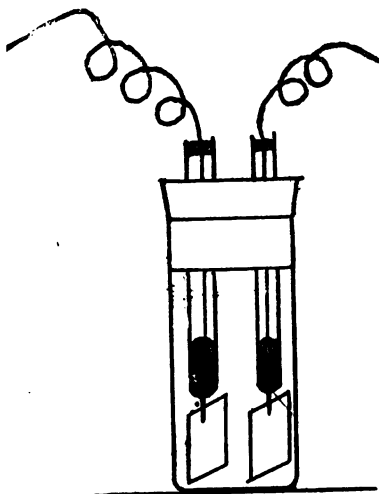


Fig. 13.

tested qualitatively for electrical conductivity:

Dilute hydrochloric acid,  
Concentrated hydrochloric acid,  
Distilled water,  
Tap water,  
Dilute acetic acid,  
Dilute potassium hydroxide solution,  
Dilute ammonium hydroxide,  
Cane sugar solution,  
Cupric sulphate solution,  
Sodium chloride solution,  
Sodium chloride (dry salt),  
Kerosene.

Remove from the cell the stopper carrying the platinum electrodes and introduce sufficient of the dilute hydrochloric acid to have the electrodes covered with liquid. Exercise care in handling the electrodes. Have them parallel at the outset and then do not shift or bend the plates during the measurements. Replace the electrodes and make sure that the wires leading into the glass tubes make contact with the mercury. Now close the switch for a moment and observe any action within the cell, and note the brightness of the lamp. (?) Empty the cell, rinse it and the electrodes well with water, and then continue the examination of the other materials. (?) Dry the cell before introducing the dry sodium chloride.

Arrange the substances in the order of their conductivity.

b. Perform an experiment that will throw some light on how the concentration of the dissolved substance influences the resistance of an electrolyte.

Taking the above substances as typical of the classes to which they belong, indicate those classes of compounds which readily conduct, and are therefore ionized in aqueous solution.

c. In aqueous solutions the radicals hitherto considered are the ions. (K. 428; S. 219.) Give the names and symbols of the three chief constituents in addition to water in aqueous solutions of sodium chloride, sodium hydroxide, cupric sulphate, and sulphuric acid.

Write in ionic form the equations that appear in your notebook for the experiments in the Chapter on Acids, Bases, and Salts.

What evidence can you cite in favor of the view that the ions are electrically charged particles?

Define acid and base in terms of the hypothesis of ions.

Formulate the following actions in terms of the ionic hypothesis:

The displacement of hydrogen from dilute acids by zinc.

The displacement of iodine from solutions of iodides by chlorine.

The displacement of sulphur from hydrogen sulphide by iodine.

The precipitation of cupric sulphide by the reaction of hydrogen sulphide with a solution of cupric sulphate.

## CHAPTER XIII.

### ELECTIVE QUALITATIVE AND QUANTITATIVE EXPERIMENTS.

#### 46. Volume of Hydrogen from Hydrogen Chloride.

Prepare a small quantity of sodium amalgam by taking 10 cc. of dry mercury in a dry crucible and adding small pieces of sodium until about 2 g. have been used. The sodium should be clean and freshly cut. The pieces are taken up with the forceps and then thrust below the surface of the mercury. The preparation of the amalgam should be conducted under a hood. Keep the amalgam in a dry test tube provided with a stopper until wanted for use. Prepare dry hydrogen chloride and fill a dry tube about 60 cm. long and 15 mm. diameter (storeroom) with the gas by displacing the air from it by means of a long delivery tube passing to the bottom. Allow the gas to pass for some time to thoroughly displace the air. When the tube is filled, withdraw the delivery tube slowly. Pour into it about half of the sodium amalgam, close quickly and tightly with the thumb slightly moist, and shake to decompose the hydrogen chloride.

Invert the tube in a basin of water and withdraw the thumb. Now transfer to a tall cylinder of water, equalize the water levels and mark the volume of the remaining gas with a tight rubber band. Measure the volume of the tube and the volume of the residual gas. Correct the volume of hydrogen remaining for aqueous tension, and then compare its volume with the original gas. Return the mercury to the shelf bottle of impure mercury.

Repeat the experiment with the remaining quantity of amalgam.

Explain the change in volume that has taken place.

#### 47. Volumetric Composition of Ammonia.

Fit the long tube of the previous experiment with a one-hole rubber stopper through which a short stemmed drop funnel is passed, in such a way that the end of the stem is

even with the end of the cork. Fill the dry tube with chlorine as it was filled in the last experiment with hydrogen chloride. Having the stop cock of the drop funnel closed, fill the short stem with water and fix the cork in the tube. Pour into the funnel about 5 cc. of ammonium hydroxide, and let most of it gradually enter the tube. When the reaction is complete, flow in dilute sulphuric acid to neutralize the excess of ammonium hydroxide in the tube. At no time during these operations must air be allowed to enter through the funnel. Finally admit through the funnel as much of dilute acid or water as will enter the tube. Measure the volume of the residual gas in the tube and compare it with the volume of the tube.

What has become of the chlorine that filled the tube? What is the residual gas? From what substance did it come and what volume would this substance occupy in the free state?

**48. Qualitative Separation of Lead, Silver, Mercurous, and Potassium Ions.**

a. In separate test tubes place about 10 cc. of the following solutions: Lead nitrate, silver nitrate, mercurous nitrate and potassium nitrate. Add dilute hydrochloric acid to each until the action is complete. (?) Allow the precipitates to settle and then pour off the supernatant liquids from each of the tubes. Add to the precipitates enough cold water nearly to fill the tubes, and shake the contents. After the precipitates have settled, decant the liquids. What compound has largely been removed by these operations? Try next the effect of hot water on the precipitates. (?) Try the effect of ammonium hydroxide on the precipitates. (?)

b. Mix about 5 cc. of each of the above solutions of nitrates. To the mixture add dilute hydrochloric acid until precipitation is complete. Of what does the precipitate consist? Filter off the precipitate. What basic ion is now in the filtrate? Wash the precipitate on the filter with a little cold water, and reject the washings. Next wash the precipitate with hot water, keeping the washings. Divide the filtrate into three parts; to one add a few drops of dilute sulphuric acid; to another a few drops of potassium chromate solution, and to the third add a little hydrogen sulphide water, or pass in a few

bubbles of hydrogen sulphide. The tests are taken as confirmatory evidence for the presence of lead ion in the filtrate. Write the equations for the confirmatory tests.

Wash the precipitate on the filter with hot water until the washings are free from lead. How is this determined? Reject the washings. Now wash the precipitate remaining on the filter with ammonium hydroxide, keeping the washings. (?) Acidify the filtrate with nitric acid. (?) What separation was made by washing the precipitate with ammonium hydroxide? Suggest a confirmatory test for silver chloride.

Dissolve the black precipitate on the filter in aqua regia, made by adding 1 cc. of nitric acid to 3 cc. of hydrochloric acid and warming. Dilute with water the solution thus obtained, and put into it a bright strip of copper. After several minutes, remove the strip, and wash and rub it. (?)

c. Obtain from the instructor an unknown solution that may contain one or more of the above ions. Using the methods in b, analyze the solution for lead, silver, mercury, and potassium, recording all steps of the process, even those giving negative results.

## CHAPTER XIV.

### INORGANIC PREPARATIONS.

#### 49. Silicon Tetrafluoride.

Arrange an apparatus as shown in Fig. 14. Prepare a mixture of 15 g. of finely ground sand and 10 g. of powdered fluorspar. Place the mixture in a flask of about 250 cc. capacity, and pour in enough concentrated sulphuric acid to make by shaking a thin paste of the mixture. Connect the flask with a dry exit tube which is connected with a longer delivery

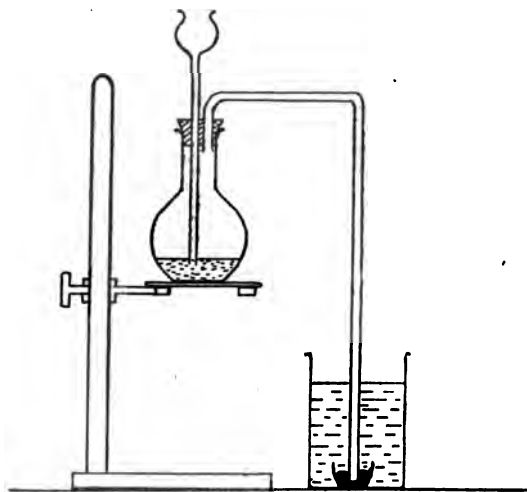


Fig. 14.

tube also dry, dipping under a little mercury contained in a small crucible, standing in the bottom of a beaker of about 200 cc. capacity. The delivery tube and mercury must be dry. Nearly fill the beaker with water without letting any of it enter the delivery tube. Heat the flask carefully, and note



the formation in the beaker of the jelly-like silicic acid. Stir occasionally, and continue to pass the gas (what is it?) until the entire liquid becomes a jelly. Disconnect the apparatus, pour the contents of the generator into the waste-jar, and rinse the flask. Remove the crucible and mercury, and pour the mercury into the bottle for impure mercury. Do not pour it into the sink or waste-jar.

Filter the contents of the beaker and save the filtrate. Wash the jelly twice on the filter, rejecting the washings. Heat a portion of it with a small volume of sodium hydroxide solution, using as little as is necessary to dissolve it. Name the solution. Add hydrochloric acid to it drop by drop. What is the precipitate that forms? Dry the rest of the precipitate by pressing it between filter papers, and then heat it gently until a white powder is obtained. (?)

Test the filtrate with blue litmus paper. (?) To one portion add a solution of barium chloride, (?) and to another potassium nitrate solution. (?) Shake the precipitates and examine their structure. (?)

Explain the chemical changes involved in every step of the experiment.

#### **50. Purification of Sodium Chloride.**

Prepare about 150 cc. of a cold, saturated solution of crude salt by grinding the salt for some time in a mortar with water. Filter the solution into a beaker and pass hydrogen chloride into the solution. Prepare the gas by dropping concentrated sulphuric acid from a drop-funnel on common salt covered with concentrated hydrochloric acid in the ordinary form of generator. Deliver the gas into the solution through a funnel with the mouth downward. (?) After considerable precipitation has occurred filter off the salt crystals. Wash once with distilled water, and remove some of the capillary water by means of a suction filter. Transfer the salt to a clean evaporating dish and dry by gentle heating. Reserve about 5 g. of the product for some tests and turn over the balance to the instructor.

Compare the purity of the product with the original sample of salt by making the following qualitative tests:

- (a) To solutions of each, add sodium carbonate solution. (?)
- (b) Test solutions of each for the sulphate ion.
- (c) Test solutions of each for the magnesium ion. (K. 396; S. 430.) (Instructions).

#### 51. Anhydrous Ferric Chloride.

In a hard, glass tube at least 30 cm. long, place several coils of bright, iron wire. Fit one end of the tube with a one-hole cork and short, glass tube and mount the tube for heating as in Exp. 7b. Connect the tube with an apparatus for generating dry chlorine (how is the gas dried?) and push the iron wire toward the end of the tube connected with the generator. Pass a gentle stream of chlorine over the iron and heat the latter moderately. Vapors of ferric chloride will soon appear and condense in the cool portion of the tube. Absorb the excess chlorine in caustic soda solution. After some time when a fair quantity of sublimate has formed, and while the gas is still passing, place a clean dry test tube over the open end of the tube and sublime the ferric chloride into the test tube by careful manipulation of the flame. Preserve the crystalline sample of ferric chloride thus obtained by sealing off the tube quickly in the blast lamp.

Test small portions of the salt remaining in the tube as follows:

- (a) Prepare a solution of the salt and divide it into four portions. To one portion add ammonium hydroxide and boil. (?)
- (b) Add a drop of the solution to 5 cc. of ammonium thiocyanate solution. (?)
- (c) To the third portion add a few drops of a solution of potassium ferrocyanide. (?)
- (d) To the fourth portion add a few drops of concentrated hydrochloric acid and a few small pieces of zinc. From time to time test the solution by pouring a drop of it into a little ammonium thiocyanate solution. (?)

#### 52. Sulphuryl Chloride. (Two students working together.)

Chlorine and sulphur dioxide unite when exposed to sunlight to form a liquid known as sulphuryl chloride. When chlorine

is passed into a solution of camphor in sulphur dioxide, the union takes place much more readily, and may be conducted at room temperatures. The camphor remains unchanged in the process and so exercises a catalytic effect.

Set up a generator for preparing dry chlorine, and another one for furnishing sulphur dioxide. The latter may however be conveniently taken from a drum of liquid sulphur dioxide. (See instructor.) Fit a wide-mouth bottle with a three-hole stopper provided with two L-tubes, whose lower ends extend nearly to the bottom of the bottle, in which the synthesis of the chloride is to be effected. Place about 20 g. of camphor in the bottle and set it in a dish of cold water. Now pass in slowly dry sulphur dioxide and observe that the camphor gradually liquefies. What is the boiling point of liquid sulphur dioxide? How do you explain its condensation at the higher temperature? When most of the camphor has dissolved start the stream of chlorine into the liquid, and continue passing both gases at about the same rate until the volume of the solution has increased about 25 cc. At least an hour will be required for this operation. Toward the close of the experiment discontinue the flow of sulphur dioxide, but maintain the stream of chlorine. (Object of this?) Before setting up the chlorine generator calculate the quantities of the materials to be used for preparing the chlorine for 40 g. of sulphuryl chloride.

Transfer the liquid to a dry distilling flask, connect to a condenser, and distill on a water-bath, collecting the distillate in a dry receiver. Finally rectify the distillate by a second distillation (Exp. 26), noting the temperature at which the liquid distills, and read the barometer. Weigh the final product, and estimate the cost of the chemicals used for its preparation assuming that all of the by-products are waste materials.

Pour a few drops of water into a small portion of the liquid. (?) Test the liquid resulting from the interaction for the sulphate ion.

**53. Precipitated Chalk.**

With the co-operation of the instructor secure about 500 cc. of the spent liquors from one of the Kipp generators used for the supply of carbon dioxide. Make the solution slightly alkaline by adding ammonium hydroxide, and then heat it to boiling. What substances are probably present in the precipitate? Filter off the precipitate and reject it. The filtrate should be clear and colorless. Take about 100 cc. of the filtrate, cool it to 20°, and determine its density with the hydrometer, and by referring to the table on p. 331 of the physical-chemical tables of Landolt, Börnstein, and Meyerhoffer determine the approximate quantity of calcium chloride in the entire solution you are using in the experiment. Then calculate the quantity of ammonium carbonate required to precipitate the calcium as carbonate. Dissolve this amount of ammonium carbonate in sufficient cold water to make a 10 per cent solution. Dilute the filtrate with distilled water till the volume is about 2-3 liters, and heat it to boiling in a large evaporating dish. Now gradually stir in the ammonium carbonate solution. How will you ascertain whether you have added enough? Allow the precipitate to settle, and then decant off the supernatant liquid and set it aside. What does it contain? Wash the precipitate with water made slightly alkaline with ammonium hydroxide until the chlorides are washed out. (?) Finally drain the precipitate well, dry it, and weigh it. How does the yield compare with the quantity of carbonate as calculated from the concentration of calcium chloride in the original solution? Consult a price-list of chemical reagents and make an estimate of the comparative cost of the product that you have prepared.

Recover the salt the filtrate contains.

What is the chief use of precipitated chalk?

**54. Chrome Alum.**

Dissolve 10 g. of potassium dichromate in water, and add the amount of sulphuric acid necessary to form potassium sulphate and chromium sulphate. Warm the solution and then add gradually enough alcohol to make the solution bright green. Ten cc. of 95 per cent alcohol will answer. Note the odor of the vapors that are produced. (?) (S. 478.) Set

the greater portion of the solution aside to evaporate spontaneously and hang a string in the solution. To obtain a large crystal with all faces well formed its growth should not be checked in certain directions by groups of other crystals clustering about it. Remove several of the crystals that have formed on the string by removing the intervening ones: If any crystals have formed on the bottom or sides of the vessel, transfer the solution to another vessel and filter it. (?) Again hang the string in the solution, and in this way secure several good sized and symmetrically developed crystals.

Concentrate the smaller portion of the solution on the water-bath till crystals appear. Examine the form and color of the crystals from both portions. (?) What is the color of their solution in water. (?)

Suggest a method of converting the chrome alum back into potassium dichromate.

## APPENDIX.

### I. ABBREVIATIONS.

The following abbreviations are used in this outline:—

cc. cubic centimeter (s)	mg. milligram (s)
cm. centimeter (s)	mm. millimeter (s)
g. gram (s)	l. liter (s)
kg. kilogram (s)	Quant. quantitative

K. Kahlenberg's Outlines of Chemistry.

S. Alexander Smith's General Chemistry for Colleges.

### II. Measures of Length and Equivalents.

1 meter = 10 decimeters = 100 cm. = 1000 mm. = 39.37 inches.

1 foot = 30.48 cm.; 1 in. = 2.54 cm. = 25.4 mm.

### III. Measures of Capacity.

1 liter = 1000 cc. = 1.057 quarts.

1 quart (U. S., liquid) = 946.36 cc.

1 ounce, avoirdupois = 28.35 cc.

1 pound, avoirdupois = 453.6 cc.

### IV. Measures of Weight.

1 kg. = 1000 g. = weight of 1000 cc. of water at 4° = 2.205 lbs.

1 oz. (av.) = 28.35 g.; 1 g. = 15.43 grains; 1 mg. = 0.001 g.

### V. Correction of Barometric Readings.

To reduce the reading taken at room temperatures to the corresponding height of a column of mercury at 0°, subtract the proper number in the correction column from the actual reading in millimeters.

Temp.	Corr'n.	Temp.	Corr'n.	Temp.	Corr'n.	Temp.	Corr'n.
12	1.6	17	2.2	22	2.85	27	3.5
13	1.7	18	2.3	23	3.0	28	3.6
14	1.8	19	2.5	24	3.1	29	3.75
15	2.0	20	2.6	25	3.2	33	4.15
16	2.1	21	2.7	26	3.35	35	4.55

**VI. Pressure of Water Vapor, or Aqueous Tension, in mm. of Mercury.**

Temp.	Press.	Temp.	Press.	Temp.	Press.
0°	4.6	19	16.4	29	29.8
5	6.5	20	17.4	30	31.55
10	9.2	21	18.5	31	33.4
12	10.5	22	19.7	32	36.4
13	11.2	23	20.9	33	37.4
14	11.9	24	22.2	34	39.6
15	12.7	25	23.55	35	41.85
16	13.55	26	25.0	40	55.0
17	14.45	27	26.5	60	149.2
18	15.4	28	28.1	100	760.0

**VII. Reduction of a Gas Volume to Standard Conditions.**

Assuming a volume of air standing over water measures 400 cc. when the barometer reads 742 mm. and the temperature is 27° C., what would be the volume of the air at standard conditions, 0° C. and 760 mm.?

If the temperature near the barometer is 27°, the column of mercury is longer than it would be at 0°. The correction to be deducted appears in Appendix 5 as 3.5 mm. Hence the corrected barometric reading is 742—3.5 or 738.5 mm.

The gas volume measured is not dry air, but a mixture of air and water vapor, whose combined pressure is 738.5 mm. By referring to Appendix 6, we see that the partial pressure of the water vapor at 27° is 26.5 mm. Hence the partial pressure of the air is 738.5—26.5 or 712 mm., which is equivalent to saying that the air when dry and in a space of 400 cc. would exert a pressure of 712 mm.

What would be the volume of the air if the pressure were increased to 760 mm.? The increase in pressure compresses the gas, and while the temperature is kept constant, the volume varies inversely as the pressure. (Boyle's law.) The volume will then change in the ratio of 712 to 760, or the volume under a pressure of 760 mm. and 27° is given by  $400 \times \frac{712}{760}$ .

Now lowering the temperature to  $0^{\circ}$ , another contraction in volume occurs. Since the volume of a gas varies directly as the absolute temperature, the pressure remaining constant, (Law of Charles) and the temperatures here considered are  $273^{\circ}$  absolute ( $= 0^{\circ}$  C.) and  $300^{\circ}$  Abs. ( $= 27^{\circ}$  C.), the reduction factor is  $\frac{273}{300}$ . The complete equation for reduction of gas to standard conditions then becomes

$$400 \times \frac{760}{760} \times \frac{273}{300} = 354.9 \text{ cc.}$$

The factors in this equation are the observed volume,  $V$ , the partial pressure,  $P$ , the standard pressure  $P_0$ , the standard temperature,  $T_0$  the observed temperature,  $T$ , and the volume reduced to standard conditions,  $V_0$ . We may then write the general equation for the reduction of a gas to standard conditions in the form,—

$$V_0 = \frac{V P T_0}{P_0 T}$$

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8. Solubility of Bases and Salts at 18°

	Fl	Cl	Br	I	NO <sub>3</sub>	SO <sub>4</sub>	CO <sub>3</sub>	OH	S
K	92.56	32.95	65.86	137.5	30.34	11.11	108	142.9	90.1
Na	4.44	35.86	88.76	177.9	83.97	16.83	19.39	116.4	17
NH <sub>4</sub>	S	36.4	73	167	180	75	S	256	S
Ag	195.4	0.00016	0.00001	0.0435	213.4	0.55	0.003	0.01	0.0132
Ba	0.16	37.24	103.6	201.4	8.74	0.00023	0.0023	3.7	I
Sr	0.012	51.09	96.52	169.2	66.27	0.011	0.0011	0.77	I
Ca	0.0016	73.19	143.3	200	121.8	0.2	0.0013	0.17	I
Mg	0.0076	55.81	103.1	148.2	74.31	35.43	0.1	0.001	I
Zn	0.005	203.9	478.2	419	117.8	53.12	0.004	0.001	I
Pb	0.07	1.49	0.6	0.08	51.66	0.004	0.0001	0.01	0.0001

The numbers express the grams of the anhydrous salt dissolved in 100 g. of water.

S indicates that the salt is soluble in water; I, that it is but very slightly soluble.







